# Attachment H

## Laboratory Validation Packages

H-1 AXYS Laboratory Validation Packages (Environmental Samples)
H-2 AXYS Laboratory Validation Packages (SAFF® Samples)
H-3 AXYS Laboratory Validation Packages (DE-FLUORO™ Samples)
H-4 ALS Laboratory Validation Packages

# Attachment H-1
# AXYS Laboratory Validation Packages (Environmental Samples)

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG69202 Report date (mm/dd/yyyy): 08/07/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☐ | ☒ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA water limits used: 70% to 130% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | Not requested per COC |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☐ | ☐ | ☒ | No QC excceedances |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Review using MPCA Guidance on Perfluorochemicals Analysis (July 2020). No data validation qualifiers.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/14/2020

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70364 Report date (mm/dd/yyyy): 11/18/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/14/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | One of the COCs was not signed by the laboratory (p 72). |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | One of the cooler was received at 7.1 °C. Associated sample results were qualified as estimated (UJ and J-). See Section 10 and data qualification summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedance as discussed in 1C. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs (trip blanks). |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | EQ-CREW-A-081319 (L31717-31), EQ-CREW-B-081319 (L31717-32), Q-CREW-B-081419 (L31727-2); EQ-CREW-A-081419 (L31731-5), EQ-CREW-A-081519 (L31731-6), EQ-CREW-B-081519 (L31731-7)<br>Results for 5 trip blanks reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects for EQ-CREW-B-081319 (L31717-32):<br>PFTrDA 0.809 J ng/L<br>PFOS 0.930 J ng/L<br>6:2 FTS 15 ng/L<br>Associated with shovel used to collect sediment samples RC10, RC11, RC12, and RC17 collected on 8/12/19 (reported in DPWG70394; see DPWG70394 for qualifications).<br>814819993217 (L31731-3) Trip Blank received 8/16/18 10:35:<br>PFOS at 0.876 J ng/L<br>Associated samples reported in DPWG70388. Associated sample concentrations were greater than 5 times the blank concentration and were acceptable without qualification. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Associated samples qualified in DPWG70394 |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | Yes | No | N/A | Comments |
|---|---|---|---|---|

| | | | | | | |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary for a listing of exceedances and qualifications. |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | Results were qualified as estimated (UJ/J). |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | ☒ | ☐ | ☐ | MS/MSD: RC2-WAT-SUR-03-081519 |
| | i. | Have the required matrix spikes been prepared and reported? | ☒ | ☐ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | ☒ | ☐ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130% See Section 10 and data qualifications summary. |
| | vi. | If yes, did the lab re-run an MS/MSD? | ☐ | ☒ | ☐ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD and LCSD; FD (EP12-WAT-SUR-02-081319, L31717-28) |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (for FDs 30%, or ± RL for results within 5 times the RL) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/14/2020

## Data Validation Qualifications for DPWG70364

**Chain of custody preservation, and holding time (Section 1C):** Results for sample RC18-WAT-SUR-01-081319 (L31717-30) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.1 °C.

**Blanks (Section 3Aii):** Detects for EQ-CREW-B-081319 (L31717-32):

PFTrDA 0.809 J ng/L
PFOS 0.930 J ng/L
6:2 FTS 15 ng/L

Associated with shovel used to collect 8/12/19 sediment samples RC10, RC11, RC12, and RC17 (reported in DPWG70394; see DPWG70394 for qualification).

814819993217 (L31731-3) Trip Blank received 8/16/18 10:35:

PFOS at 0.876 J ng/L

Associated samples reported in DPWG70388. Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9-WAT-18-21-01-081319 | L31717-18 | C13-PFBA | 6.89 | J+ |
| EP14-WAT-SUR-01-081319 | L31717-19 | C13-PFBA | 5.93 | J+ |
| EP14-WAT-18-21-01-081319 | L31717-20 | C13-PFBA | 20.0 | J+ |
| EP15-WAT-SUR-01-081319 | L31717-21 | C13-PFBA | 9.26 | J+ |
| EP15-WAT-18-21-01-081319 | L31717-22 | C13-PFBA | 9.87 | J+ |
| WL11-WAT-SUR-01-081319 | L31717-24 | C13-PFBA | 12.4 | J+ |
| EP11-WAT-SUR-01-081319 | L31717-25 | C13-PFBA | 35.3 | J+ |
| EP10-WAT-SUR-01-081319 | L31717-26 | C13-PFBA | 33.1 | J+ |
| EP12-WAT-SUR-01-081319 | L31717-27 | C13-PFBA | 6.34 | J+ |
| EP12-WAT-SUR-02-081319 | L31717-28 | C13-PFBA | 6.16 | J+ |
| WL17-WAT-SUR-01-081319 | L31717-29 | C13-PFBA | 7.97 | J+ |
| RC1-WAT-18-21-01-081519 | L31727-3 | C13-PFBA | 7.14 | J+ |
| RC1-WAT-SUR-01-081519 | L31727-4 | C13-PFBA | 5.50 | J+ |
| RC2-WAT-SUR-03-081519 | L31727-5 (A) | C13-PFBA | 9.40 | J+ |
| RC2-WAT-SUR-02-081519 | L31727-7 | C13-PFBA | 14.3 | J+ |
| RC2-WAT-18-21-01-081519 | L31727-8 | C13-PFBA | 23.7 | J+ |
| 814819993099 | L31731-4 | D3-N-MeFOSA | 48.2 | UJ |
| RC3-WAT-SUR-01-081419 | L31731-8 | C13-PFBA | 7.63 | J+ |
| RC7-WAT-SUR-01-081419 | L31731-9 | C13-PFBA | 5.15 | J+ |
| RC11-WAT-SUR-01-081419 | L31731-11 | C13-PFBA | 8.92 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-WAT-SUR-01-081419 | L31731-12 | C13-PFBA | 3.76 | J+ |
| RC17-WAT-SUR-01-081419 | L31731-14 | C13-PFBA | 5.38 | J+ |
| WL1-WAT-SUR-01-081519 | L31731-17 | C13-PFBA | 8.54 | J+ |
| WL2-WAT-SUR-01-081519 | L31731-18 | C13-PFBA | 8.90 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **RC2-WAT-SUR-03-081519:** | | | |
| 6:2 FTS | **56.1/57.7** | 70-130 | The detect for sample RC2-WAT-SUR-03-081519 were qualified as estimated biased low (J-). |
| PFBA | **23.9/14.8** | 70-130 | Since both high bias (due to labeled analog recovery) and low bias (due to MS/MSD recoveries) are present, this result was qualified as estimated (J). |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

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*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70388 Report date (mm/dd/yyyy): 11/18/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/28/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | The COC for sample WL13-WAT-SUR-01-081419 (L31731-30) was not signed by the laboratory (p 32). |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | One of the coolers was received at 7.4 °C. Associated sample results were qualified as estimated (UJ and J-). See Section 10 and data qualifications summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedance as discussed in 1C. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | The trip blank 814819993217 (L31731-3), reported in DPWG70364, had the following detect:<br>PFOS at 0.876 J ng/L<br>(associated with samples L31731-25, -26, -31, -33, -34, -35)<br>The trip blank 814819993099 ( L31731-4) reported in DPWG7036 was nondetect. This trip blank was associated with WL13-WAT-SUR-01-081419 (L31731-30): |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Associated sample concentrations for trip blank 14819993217 ( L31731-3) were greater than 10 times the blank concentration and were acceptable without qualification. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than $1/10^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | See Section 10 and the qualification summary for a listing of exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | Results were qualified as estimated (UJ and J). |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | Not requested per COC |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LSCD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD, FD (WL9-WAT-SUR-02-081419, L31731-26) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/28/2020

## Data Validation Qualifications for DPWG70388

**Chain of custody preservation, and holding time (Section 1C):** Results for sample WL9-WAT-SUR-01-081419 (L31731-25), WL9-WAT-SUR-02-081419 (L31731-26), WL14-WAT-SUR-01-081419 (L31731-31), and WL16-WAT-SUR-01-081419 (L31731-33) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.4 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL7-WAT-SUR-01-081419 | L31731-23 | 13C4-PFBA | 9.24 | J+ |
| WL9-WAT-SUR-01-081419 | L31731-25 | 13C4-PFBA | 18.9 | J+ |
| | | 13C2-PFTeDA | 46.2 | UJ |
| WL9-WAT-SUR-02-081419 | L31731-26 | 13C4-PFBA | 38.5 | J+ |
| | | D3-N-MeFOSA | 39.6 | UJ |
| | | D5-N-EtFOSA | 41.1 | UJ |
| WL10-WAT-SUR-01-081419 | L31731-28 | 13C2-PFTeDA | 41.2 | UJ |
| | | d9-NEt-FOSE | 45.2 | UJ |
| WL13-WAT-SUR-01-081419 | L31731-30 | 13C4-PFBA | 4.99 | J+ |
| | | 13C2-PFTeDA | 46.8 | UJ |
| WL14-WAT-SUR-01-081419 | L31731-31 | 13C4-PFBA | 4.51 | J+ |
| WL16-WAT-SUR-01-081419 | L31731-33 | 13C4-PFBA | 4.53 | J+ |
| WL18-WAT-SUR-01-081519 | L31731-34 | 13C4-PFBA | 3.71 | J+ |
| WL19-WAT-SUR-01-081519 | L31731-35 | 13C4-PFBA | 39.4 | J+ |

Results with both high bias (due to labeled analog recovery) and low bias (due to receipt temperature), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70390 Report date (mm/dd/yyyy): 11/18/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/4/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Some of the samples were received at elevated temperatures. Trip blank sample (814819993309) was received at 12.7 °C. Sample RC7-FOAM-081219 (L31822-5) was received at 6.5 °C. Associated sample results were qualified as estimated (UJ and J-). See Section 10 and data qualification summary. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip Blank: 814819993309 (not associated with foam samples, associated with samples collected 8/27/19). |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | See Section 10 and the qualification summary for a listing of exceedances and qualifications. |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | Results were qualified as estimated (UJ). |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | Recovery for 6:2 FTS is 139%. See Section 10 and the summary of qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | Not requested per COC |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LSCD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | 30% limit for PFAS. 6:2 FTS - 39.8% RPD. Associated detects previously qualified J+ due to high LCS recovery. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Elevated reporting limits due to high sample concentrations.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/4/2020

## Data Validation Qualifications for DPWG70390

**Chain of custody preservation, and holding time (Section 1C):** Results for samples 814819993309 (L31815-1) and RC7-FOAM-081219 (L31822-5) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 12.7 and 6.5 °C, respectively.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| 814819993309 | L31815-1 | D3-N-MeFOSA | 49.1 | UJ |

**LCS/LCSD (Section 5Bi):** Detects for 6:2 FTS were qualified as estimated biased high (J+) and include samples RC7A-FOAM-081419 (L31822-1) and RC12-FOAM-081419 (L31822-2).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70394 Report date (mm/dd/yyyy): 11/7/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/4/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at elevated temperatures. See Section 10 and data qualifications summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used - ok |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Equipment Blank EQ-CREW-B-081319 (L31717-32, reported in DPWG70364):<br>PFTrDA 0.809 J ng/L<br>PFOS 0.930 J ng/L<br>6:2 FTS 15 ng/L<br>Associated with shovel used to collect sediment samples RC10, RC11, RC12, and RC17 collected on 8/12/19. See data qualifications summary for qualifications.<br>L31727-2 (EB) reported in DPWG70364 - ND<br>L31731-5 (EB) reported in DPWG70364 - ND<br>Associated trip blanks:<br>L31697-3 not reported<br>L31727-1 reported in DPWG70364 - ND<br>L31731-4 reported in DPWG70364 - ND<br>L31717-17 not reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Listed above (A) |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | Yes | No | N/A | Comments |
|---|---|---|---|---|

| | | | | | | |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | Results were qualified as estimated (UJ and J). See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS sed/other limits used: 50% to 150%. N-MeFOSA and N-EtFOSA reported as not quantifiable (NQ). Most N-MeFOSA and N-EtFOSA results were qualified due to labeled analogs. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | ☒ | ☐ | ☐ | WL9-SED-0-6-03-081419; EP12-SED-0-6-03-081319 |
| | ii. | If no, is there and explanation in the report as to why? | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | ☐ | ☒ | ☐ | MPCA PFAS sed/other limits used: 50% to 150% |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSDs; FDs: RC3-SED-0-6-02-081219, RC17-SED-0-6-02-081219, WL9-SED-0-6-02-081419, WL9-SED-0-6-03-081419, EP12-SED-0-6-02-081319, EP12-SED-0-6-03-081319 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 50% limit for PFAS (sed). See Section 10 and data qualifications summary for exceedances. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | Samples dried prior to prep. Reported for V69529 samples only. |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Some of the labeled analog low recoveries were not flagged. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/9/2020

## Data Validation Qualifications for DPWG70394

**Chain of custody preservation, and holding time (Section 1C):** Results for samples RC3-SED-0-6-01-081219 (L31707-1), RC3-SED-0-6-02-081219 (L31707-2), RC4-SED-0-6-01-081219 (L31707-3), RC5-SED-0-6-01-081219 (L31707-4), RC6-SED-0-6-01-081219 (L31707-5), RC7-SED-0-6-01-081219 (L31707-6), RC8-SED-0-6-01-081219 (L31707-7) and RC9-SED-0-6-01-081219 (L31707-8) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 6.5 °C.

Results for samples RC11-SED-0-6-01-081219 (L31707-10), RC12-SED-0-6-01-081219 (L31707-11), and RC15-SED-0-6-01-081219 (L31707-14) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 6.9 °C.

Results for samples RC1-SED-0-6-01-081519 (L31729-1) and RC2-SED-0-6-01-081519 (L31729-2) were nondetect and qualified as estimated (UJ) due to a sample receipt temperature of 13 °C.

Results for samples WL13-SED-0-6-01-081419 (L31730-8), WL2-SED-0-6-01-081519 (L31730-11) and EP13-SED-0-6-01-081519 (L31730-12) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.2 °C.

Results for samples WL9-SED-0-6-01-081419 (L31730-4), WL9-SED-0-6-02-081419 (L31730-5), WL9-SED-0-6-03-081419 (L31730-6), WL14-SED-0-6-01-081419 (L31730-9), and WL15-SED-0-6-01-081419 (L31730-10) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.4 °C.

Results for samples WL17-SED-0-6-01-081319 (L31739-8) and RC18-SED-0-6-01-081319 (L31739-9) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.1 °C.

**Blanks (Section 3Aii):** EQ-CREW-B-081319 (L31717-32; reported in DPWG70364) associated with shovel used to collect sediment samples RC10, RC11, RC12, and RC17 collected on 8/12/19:

PFTrDA 0.809 J ng/L
PFOS 0.930 J ng/L
6:2 FTS 15 ng/L

PFOS results for samples RC10-SED-0-6-01-081219, RC17-SED-0-6-01-081219 and RC17-SED-0-6-02-081219 were within 10 times the blank concentration and were qualified B. Associated PFTrDA and 6:2 FTS results were nondetect and were acceptable without qualification.

**Labeled Analogs -surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC4-SED-0-6-01-081219 | L31707-3 | D3-N-MeFOSA | 30.6 | J+ |
| | | D5-N-EtFOSA | 18.5 | J+ |
| | | d7-NMe-FOSE | 46.3 | UJ |
| | | d9-NEt-FOSE | 32.3 | UJ |
| RC5-SED-0-6-01-081219 | L31707-4 | D3-N-MeFOSA | 31.3 | J+ |
| | | D5-N-EtFOSA | 28.2 | J+ |
| RC6-SED-0-6-01-081219 | L31707-5 | D3-N-MeFOSA | 21.0 | UJ |
| | | D5-N-EtFOSA | 19.3 | J+ |
| | | d7-NMe-FOSE | 45.2 | UJ |
| | | d9-NEt-FOSE | 42.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC8-SED-0-6-01-081219 | L31707-7 | D5-N-EtFOSA | 48.4 | UJ |
| RC9-SED-0-6-01-081219 | L31707-8 | D3-N-MeFOSA | 49.8 | UJ |
| | | D5-N-EtFOSA | 44.1 | UJ |
| RC13-SED-0-6-01-081219 | L31707-12 | D3-N-MeFOSA | 47.5 | UJ |
| | | D5-N-EtFOSA | 44.4 | UJ |
| RC14-SED-0-6-01-081219 | L31707-13 | D3-N-MeFOSA | 49.4 | UJ |
| | | D5-N-EtFOSA | 46.1 | UJ |
| RC15-SED-0-6-01-081219 | L31707-14 | D5-N-EtFOSA | 49.9 | UJ |
| RC1-SED-0-6-01-081519 | L31729-1 | D3-N-MeFOSA | 46.7 | UJ |
| | | D5-N-EtFOSA | 43.7 | UJ |
| RC2-SED-0-6-01-081519 | L31729-2 | 13C4-PFBA | 16.2 | J+ |
| | | D3-N-MeFOSA | 42.8 | UJ |
| | | D5-N-EtFOSA | 36.9 | UJ |
| | | d7-NMe-FOSE | 49.3 | UJ |
| | | d9-NEt-FOSE | 39.5 | UJ |
| WL5-SED-0-6-01-081419 | L31730-1 | D3-N-MeFOSA | 43.6 | UJ |
| | | D5-N-EtFOSA | 41.4 | UJ |
| | | d9-NEt-FOSE | 36.4 | UJ |
| WL6-SED-0-6-01-081419 | L31730-2 | D5-N-EtFOSA | 47.9 | UJ |
| WL7-SED-0-6-01-081419 | L31730-3 | D3-N-MeFOSA | 40.3 | UJ |
| | | D5-N-EtFOSA | 39.5 | UJ |
| WL9-SED-0-6-03-081419 | L31730-6 (A) | D5-N-EtFOSA | 25.5 | UJ |
| | | D3-N-MeFOSA | 20.6 | UJ |
| | | d7-NMe-FOSE | 43.0 | UJ |
| | | d9-NEt-FOSE | 39.7 | UJ |
| WL12-SED-0-6-01-081419 | L31730-7 | D5-N-EtFOSA | 46.2 | UJ |
| WL13-SED-0-6-01-081419 | L31730-8 | D3-N-MeFOSA | 49.0 | UJ |
| | | D5-N-EtFOSA | 48.9 | UJ |
| WL2-SED-0-6-01-081519 | L31730-11 | D5-N-EtFOSA | 44.1 | UJ |
| EP13-SED-0-6-01-081519 | L31730-12 | 13C4-PFBA | 37.3 | J+ |
| | | D3-N-MeFOSA | 34.1 | UJ |
| | | D5-N-EtFOSA | 27.7 | UJ |
| | | d7-NMe-FOSE | 46.0 | UJ |
| | | d9-NEt-FOSE | 40.9 | UJ |
| EP4-SED-0-6-01-081319 | L31739-1 | D5-N-EtFOSA | 48.0 | UJ |
| EP7-SED-0-6-01-081319 | L31739-2 | D3-N-MeFOSA | 46.7 | UJ |
| | | D5-N-EtFOSA | 46.3 | UJ |
| WL10-SED-0-6-01-081319 | L31739-6 | D5-N-EtFOSA | 45.1 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18-SED-0-6-01-081319 | L31739-9 | 13C4-PFBA | 35.2 | UJ |
| | | D3-N-MeFOSA | 42.2 | UJ |
| | | D5-N-EtFOSA | 35.7 | UJ |
| | | d9-NEt-FOSE | 37.5 | UJ |

**Estimated Maximum Possible Concentration (EMPC):**

RC4-SED-0-6-01-081219 (L31707-3): The PFTrDA result was reported as an EMPC. This result is was qualified as estimated (J).

**Field Duplicate Precision (6Bi):**

Results qualified as estimated (J) due to field precision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **RC3-SED-0-6-01-081219/RC3-SED-0-6-02-081219:** | | | |
| PFOS | 5.89 | 14 | 82.6 |
| PFOSA | 1.71 | 3.04 | 56.0 |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70395 Report date (mm/dd/yyyy): 11/5/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/18/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Some of the samples were received at elevated temperatures. See Section 10 and data qualifications summary. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used - ok |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Not reported in this data set. Associated trip blanks are L31717-2 and L31815-1. Results were not reported for L31717-2. L31815-1 was reported in DPWG70390 and was nondetect. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | Associated results were nondetect and qualified as estimated (UJ). |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP9-SED-0-6-03-082719 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | MPCA PFAS sed/other limits used: 50% to 150% |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSDs: EP9-SED-0-6-03-082719; FD: EP9-SED-0-6-02-082719/EP9-SED-0-6-03-082719 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (sed). RPDs ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Some of the sample sizes were less than 5 grams, and limits were somewhat elevated. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | Samples dried prior to prep. |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☐ | ☒ | ☐ | Some of the labeled analog low recoveries were not flagged. |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2020

## Data Validation Qualifications for DPWG70395

**Chain of custody preservation, and holding time (Section 1C):** Results for all samples except EP5-SED-0-6-01-081319 (L31739-11) were qualified as estimated (UJ and J-) due to sample receipt temperatures of 7.1 °C and 12.7 °C. The receipt temperature for sample SED-0-6-01-081319 (L31739-11) was 3.7 °C and was acceptable.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC19-SED-0-6-01-081319 | L31739-10 | D3-N-MeFOSA | 48.7 | UJ |
| | | D5-N-EtFOSA | 48.0 | UJ |
| EP5-SED-0-6-01-081319 | L31739-11 | D5-N-EtFOSA | 48.1 | UJ |
| EP2-SED-0-6-01-082719 | L31814-2 | D5-N-EtFOSA | 47.7 | UJ |
| EP1-SED-0-6-01-082719 | L31814-3 | D3-N-MeFOSA | 43.9 | UJ |
| | | D5-N-EtFOSA | 43.3 | UJ |
| EP3-SED-0-6-01-082719 | L31814-4 | D3-N-MeFOSA | 20.2 | UJ |
| | | D5-N-EtFOSA | 17.2 | UJ |
| | | d7-NMe-FOSE | 36.3 | UJ |
| | | d9-NEt-FOSE | 30.0 | UJ |
| EP15-SED-0-6-01-082719 | L31814-5 | 13C2-PFTeDA | 46.8 | UJ |
| | | D3-N-MeFOSA | 26.2 | UJ |
| | | D5-N-EtFOSA | 21.7 | UJ |
| | | d7-NMe-FOSE | 38.1 | UJ |
| | | d9-NEt-FOSE | 30.8 | UJ |
| EP9-SED-0-6-01-082719 | L31814-7 | 13C2-PFTeDA | 48.1 | UJ |
| | | D3-N-MeFOSA | 19.4 | UJ |
| | | D5-N-EtFOSA | 14.8 | UJ |
| | | d7-NMe-FOSE | 31.5 | UJ |
| | | d9-NEt-FOSE | 26.4 | UJ |
| EP9-SED-0-6-02-082719 | L31814-8 | 13C2-PFTeDA | 43.3 | UJ |
| | | D3-N-MeFOSA | 20.0 | UJ |
| | | D5-N-EtFOSA | 17.3 | UJ |
| | | d7-NMe-FOSE | 31.2 | UJ |
| | | d9-NEt-FOSE | 26.5 | UJ |
| EP9-SED-0-6-03-082719 | L31814-9 | 13C2-PFTeDA | 42.3 | UJ |
| | | D3-N-MeFOSA | 17.8 | UJ |
| | | D5-N-EtFOSA | 18.9 | UJ |
| | | d7-NMe-FOSE | 30.7 | UJ |
| | | d9-NEt-FOSE | 25.5 | UJ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70397 Report date (mm/dd/yyyy): 11/20/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Sample SC2-WAT-18-21-01-081219 (L31697-20) was received at 6.9 °C. Results were nondetect and qualified UJ. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | 10/17/19 13:20:<br>9Cl-PF3ONS 69.3%<br>MN limits are 70% to 130% for CCVs. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | No field or trip blanks reported in this SDG. L31697-3 (Trip Blank) associated with sample RC8-WAT-SUR-01-081219 (L31697-10 RM) per COC. Results not reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | Not in this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water). ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples were prepped using reduced volume. This is ok for sample RC8-WAT-SUR-01-081219 (L31697-10 RM) as elevated concentrations were reported. Results for sample SC2-WAT-18-21-01-081219 (L31697-20) were nondetect for all analytes and may have not met reporting objectives. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/21/2020

## Data Validation Qualifications for DPWG70397

**Chain of custody preservation, and holding time (Section 1C):** Results for samples SC2-WAT-18-21-01-081219 (L31697-20) were nondetect and qualified as estimated (UJ) due to a sample receipt temperature of 6.9 °C.

**Calibration (Section 2): The** 9Cl-PF3ONS result for sample RC8-WAT-SUR-01-081219 (L31697-10 RM) was nondetect and qualified as estimated (UJ) due to a CCV exceedance.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70406 Report date (mm/dd/yyyy): 11/20/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/18/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The field sample ID was updated. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | ICAL for low level CS1 standard outside limits of 70% to 130%:<br>PFNS 67.5%<br>MeFOSAA 64.2%<br>Results were nondetect and qualified UJ.<br>Calibration verification 10/29/19 17:13:<br>PFUnA 132% - the sample result was nondetect, no qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | No field or trip blanks reported in this SDG. L31717-4 (Trip Blank) associated with sample WL11-SED-0-6-01-081319 (L31739-7 R) per COC. Results not reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 0.656 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | WL11-SED-0-6-01-081319:<br>D5-N-EtFOSA 49.6% |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | See data qualifications summary for discussion of labeled analogs for LCSD. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | Not in this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water). ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2020

## Data Validation Qualifications for DPWG70406

**Calibration (Section 2A):** Results associated with low recoveries of PFNS and MeFOSAA (all samples - WL11-SED-0-6-01-081319) were nondetect and were qualified as estimated (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL11-SED-0-6-01-081319 | L31739-7 R | D5-N-EtFOSA | 49.6 | UJ |

**Laboratory Control Sample Duplicate (Section 5Bii):** Labeled analog recoveries for the LCSD for D3-N-MeFOSA and D5-N-EtFOSA were below 10% (LCS was ok). Sample results were qualified based the sample specific labeled analog recoveries as they are more representative.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG70461 Report date (mm/dd/yyyy): 11/20/2019

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/18/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | ICAL for low level CS1 standard outside limits of 70% to 130%:<br>8:2 FTS 131% - sample results ND, no qualifiers<br>PFNS 67.5%<br>MeFOSAA 64.2%<br>PFNS and MeFOSAA results were nondetect and qualified UJ. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | No field or trip blanks reported in this SDG. L31717-2 (Trip Blank) associated with sample EP2-WAT-SUR-01-081319 (L31717-9); L31717-1 (trip blank) associated with WL10-WAT-SUR-01-081319 (L31717-23 R), L31731-2,-3,-4 (trip blanks) associated with L31731 samples per COC.<br>Results for trip blanks L31717-1 and L31717-2 not reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | L31731-2 ,-3,-4 trip blanks reported in DPWG70364, all nondetect except -3 (PFOS 0.876 ng/L). |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | No qualifiers - associated sample concentration greater than 10 times the trip blank concentration. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | WL9-WAT-SUR-03-081419 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MSD:<br>9Cl-PF3ONS 67.3%<br>11Cl-PF3OUdS 66.0%<br>See Section 10 and data qualifications summary. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water). ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | The laboratory used reduced sample volumes which resulted in elevated reporting limts. The reduced sample volume was required due to high concentrations of PFBA and PFOS. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Case narrative discusses a number of sample prep issues. The samples were re-prepped. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2020

## Data Validation Qualifications for DPWG70461

**Chain of custody preservation, and holding time (Section 1C):** Results for sample EP2-WAT-SUR-01-081319 (L31717-9 R) were qualified as estimated (UJ and J-) due to a sample receipt temperature of 7.1 °C.

Results for samples WL9-WAT-SUR-03-081419 (L31731-27 R (A)) and WL15-WAT-SUR-01-081419 (L31731-32 R) were qualified (UJ and J-) due to a sample receipt temperature of 7.4 °C.

Results for samples WL2-WAT-18-21-01-081519 (L31731-19 R) and EP13-WAT-SUR-01-081519 (L31731-37 R) were qualified (UJ and J-) due to a sample receipt temperature of 7.2 °C.

**Calibration (Section 2A):** Results associated with low recoveries of PFNS and MeFOSAA (all samples) were nondetect and were qualified as estimated (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and were qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL9-WAT-SUR-03-081419 | L31731-27 R (A) | 13C2-PFTeDA | 44.8 | UJ |
| | | d9-NEt-FOSE | 45.2 | UJ |

**Matrix Spike Duplicate (Section 6Av):** The 9Cl-PF3ONS and 11Cl-PF3OUdS results for sample WL9-WAT-SUR-03-081419 were nondetect and qualified UJ due to low MSD recoveries (67.5% and 66.0%, respectively).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72576 Report date (mm/dd/yyyy): 6/4/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Two samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Three trip blanks were reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water): LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/14/2020

## Data Validation Qualifications for DPWG72576

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were nondetect and qualified UJ due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| 392602993191 | L33047-2 | 7.7 |
| 392602814934 | L33047-3 | 6.4 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC21-WAT-BULK-01-042320 | L33047-4 | 13C4-PFBA | 44.6 | J+ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72820 Report date (mm/dd/yyyy): 7/6/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/13/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Seventeen samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blank reported in DPWG72576: 392602613575 (L33047-1) |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | The trip blank was nondetect |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | WG72424-101 i (5/28/2): 6:2 FTS 0.361 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP16-SED-0-6-03-042720<br>VB3-SED-COMP-0-6-03-042720 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSDs<br>FDs:<br>RC16A-SED-WET-0-6-01-043020/RC16A-SED-WET-0-6-02-043020<br>VB3-SED-COMP-0-6-01-042720/VB3-SED-COMP-0-6-02-042720/VB3-SED-COMP-0-6-03-042720 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☐ | ☒ | ☐ | 50% limit for PFAS (sed):<br>MSD RPDs - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | FDs: See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/13/2020

## Data Validation Qualifications for DPWG72820

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| EP16-SED-0-6-01-042720 | L33050-30 | 6.4 |
| EP16-SED-0-6-02-042720 | L33050-31 | 6.4 |
| EP16-SED-0-6-03-042720 | L33050-32 (A) | 6.4 |
| EP16-SED-6-18-01-042720 | L33050-33 | 6.4 |
| EP16-SED-30-36-01-042720 | L33050-34 | 6.4 |
| EP20-SED-WET-0-6-01-042720 | L33050-35 | 6.4 |
| EP20-SED-WET-6-12-01-042720 | L33050-36 | 6.4 |
| EP20-SED-24-30-01-042720 | L33050-37 | 6.4 |
| EP20-SED-36-42-01-042720 | L33050-38 | 6.4 |
| EP23-SED-0-6-01-042720 | L33050-39 | 6.4 |
| EP24-SED-0-6-01-042720 | L33050-40 | 6.4 |
| BP1-SED-0-6-01-042720 | L33050-41 i | 6.4 |
| VB1-SED-COMP-0-6-01-042720 | L33050-42 i | 6.4 |
| VB3-SED-COMP-0-6-01-042720 | L33050-43 i | 6.4 |
| VB3-SED-COMP-0-6-02-042720 | L33050-44 i | 6.4 |
| VB3-SED-COMP-0-6-03-042720 | L33050-45 i (A) | 6.4 |
| EP21-SED-FOAM-0-6-01-042320 | L33050-46 i | 7.7 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) as listed below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17-SED-WET-6-12-01-042520 | L33050-21 | D3-N-MeFOSA | 41.4 | UJ |
| | | D5-N-EtFOSA | 30.9 | UJ |
| EP17-SED-0-6-01-042520 | L33050-22 | D3-N-MeFOSA | 34.2 | UJ |
| | | D5-N-EtFOSA | 22.8 | UJ |
| | | d9-NEt-FOSE | 47.4 | UJ |
| EP19-SED-WET-0-6-01-042520 | L33050-23 | D3-N-MeFOSA | 42.9 | UJ |
| | | D5-N-EtFOSA | 36.7 | UJ |
| EP19-SED-WET-6-12-01-042520 | L33050-24 | D5-N-EtFOSA | 45.8 | UJ |
| EP19-SED-24-30-01-042520 | L33050-25 | D5-N-EtFOSA | 48.4 | UJ |
| RC16A-SED-WET-0-6-01-043020 | L33050-27 | D3-N-MeFOSA | 38.6 | UJ |
| | | D5-N-EtFOSA | 31.5 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC16A-SED-WET-0-6-02-043020 | L33050-28 | D3-N-MeFOSA | 32.9 | UJ |
| | | D5-N-EtFOSA | 24.6 | UJ |
| RC16A-SED-WET-6-12-01-043020 | L33050-29 | D5-N-EtFOSA | 48.0 | UJ |
| EP20-SED-WET-0-6-01-042720 | L33050-35 | D5-N-EtFOSA | 43.2 | UJ |
| EP24-SED-0-6-01-042720 | L33050-40 | D3-N-MeFOSA | 34.4 | UJ |
| | | D5-N-EtFOSA | 26.7 | UJ |
| VB1-SED-COMP-0-6-01-042720 | L33050-42 i | D3-N-MeFOSA | 41.3 | UJ |
| | | D5-N-EtFOSA | 33.4 | UJ |
| VB3-SED-COMP-0-6-01-042720 | L33050-43 i | D3-N-MeFOSA | 42.2 | UJ |
| | | D5-N-EtFOSA | 35.9 | UJ |
| VB3-SED-COMP-0-6-02-042720 | L33050-44 i | D3-N-MeFOSA | 42.2 | UJ |
| | | D5-N-EtFOSA | 36.9 | UJ |
| VB3-SED-COMP-0-6-03-042720 | L33050-45 i (A) | D3-N-MeFOSA | 40.2 | UJ |
| | | D5-N-EtFOSA | 36.3 | UJ |

**Additional comments about the lab report (Section 10):** The EtFOSAA result for sample EP19-SED-24-30-01-042520 (L33050-25) was flagged R by the laboratory indicating the peak detected but did not meet quantification criteria, and the result reported represents the estimated maximum possible concentration. This EtFOSAA result was qualified as estimated (J).

**Field Duplicate Precision (6Bi):**

Results qualified as estimated (J) due to field precision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Triplicate Result (ng/g) | RPD (%) |
|---|---|---|---|---|
| **RC16A-SED-WET-0-6-01-043020/RC16A-SED-WET-0-6-02-043020:** | | | | |
| PFOA | 0.359 | 0.107 | 0.142 | >±2xRL |
| **VB3-SED-COMP-0-6-01-042720/ VB3-SED-COMP-0-6-02-042720/ VB3-SED-COMP-0-6-03-042720:** | | | | |
| PFBA | 1.49 | 0.740 | 0.787 | 75 |
| PFOA | 0.781 | 0.408 | 0.329 | 89 |
| PFOS | 1.37 | 0.862 | 0.852 | 50 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72829 Report date (mm/dd/yyyy): 7/6/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Eighteen samples were received at an elevated temperature. See data qualifications summary for detail regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blank reported in DPWG72576: 392602613575 (L33047-1) |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | The trip blank was nondetect |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (sed):<br>LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for sample EP17-SED-WET-0-6-01-042520 (L33050-20) resulting in elevated reporting limits.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/13/2020

## Data Validation Qualifications for DPWG72829

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| RC17-SED-BANK-EAST-0-3-01-042320 | L33050-1 | 7.7 |
| RC17-SED-BANK-EAST-3-6-01-042320 | L33050-2 | 7.7 |
| RC17-SED-BANK-WEST-0-3-01-042320 | L33050-3 | 7.7 |
| RC17-SED-BANK-WEST-3-6-01-042320 | L33050-4 | 7.7 |
| RC18-SED-COMP-0-3-01-042320 | L33050-5 | 7.7 |
| RC21-SED-COMP-0-3-01-042320 | L33050-6 | 7.7 |
| FC1-SED-0-3-01-042320 | L33050-7 | 7.7 |
| RC12-SED-12-18-01-042420 | L33050-8 | 7.7 |
| RC12-SED-30-36-01-042420 | L33050-9 | 7.7 |
| RC5-SED-0-4-01-042420 | L33050-10 | 7.7 |
| RC5-SED-4-6-01-042420 | L33050-11 | 7.7 |
| RC5-SED-22-26-01-042420 | L33050-12 | 7.7 |
| RC5-SED-WET-3-12-01-042420 | L33050-13 | 7.7 |
| RC5-SED-WET-12-18-01-042420 | L33050-14 | 7.7 |
| RC6-SED-WET-0-6-01-042420 | L33050-15 | 7.7 |
| RC6-SED-WET-12-18-01-042420 | L33050-16 | 7.7 |
| RC3-SED-WET-0-6-01-042520 | L33050-17 | 7.7 |
| RC3-SED-WET-12-18-01-042520 | L33050-18 | 7.7 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC17-SED-BANK-EAST-0-3-01-042320 | L33050-1 | D5-N-EtFOSA | 43.5 | UJ |
| RC17-SED-BANK-WEST-3-6-01-042320 | L33050-4 | D5-N-EtFOSA | 49.9 | UJ |
| RC18-SED-COMP-0-3-01-042320 | L33050-5 | D5-N-EtFOSA | 43.9 | UJ |
| RC5-SED-0-4-01-042420 | L33050-10 | 13C4-PFBA | 29.2 | J+ |
| RC5-SED-WET-3-12-01-042420 | L33050-13 | 13C4-PFBA | 21.0 | J+ |
| RC6-SED-WET-0-6-01-042420 | L33050-15 | 13C4-PFBA | 17.1 | J+ |
| RC3-SED-WET-0-6-01-042520 | L33050-17 | 13C4-PFBA | 11.1 | J+ |
| | | d9-NEt-FOSE | 43.4 | J+ |
| RC3-SED-WET-12-18-01-042520 | L33050-18 | D5-N-EtFOSA | 47.3 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17-SED-WET-0-6-01-042520 | L33050-20 | 13C4-PFBA | 14.2 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72884 Report date (mm/dd/yyyy): 7/13/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at an elevated temperature. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Assocaited trip blanks reported in DPWG72576 and DPWG73031:<br>392602993191 (L33047-2)<br>393004067424 (L33098-1) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (sed): LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for sample EP18-SED-0-6-01-051220 (L33094-1) resulting in elevated reporting limits.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/14/2020

## Data Validation Qualifications for DPWG72884

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| RC12-SED-COMP-0-6-01-042420 | L33049-1 | 7.7 |
| RC3A-SED-WET-0-6-01-042520 | L33049-2 | 7.7 |
| RC12-SED-0-6-01-042520 | L33049-3 | 7.7 |
| EP18-SED-0-6-01-051220 | L33094-1 | 9.8 |
| GLI-SED-0-6-01-051220 | L33094-2 | 9.8 |
| RC22-SED-WET-0-6-01-051420 | L33094-3 | 9.8 |
| RC22-SED-WET-6-12-01-051420 | L33094-4 | 9.8 |
| RC23-SED-WET-0-6-01-051420 | L33094-5 | 9.8 |
| RC23-SED-WET-6-12-01-051420 | L33094-6 | 9.8 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3A-SED-WET-0-6-01-042520 | L33049-2 | D3-N-MeFOSA | 33.8 | J+ |
| | | D5-N-EtFOSA | 27.0 | J+ |
| | | d9-NEt-FOSE | 43.2 | J+ |
| EP18-SED-0-6-01-051220 | L33094-1 | 13C4-PFBA | 22.7 | J+ |
| | | D5-N-EtFOSA | 42.9 | UJ |
| RC22-SED-WET-0-6-01-051420 | L33094-3 | D3-N-MeFOSA | 40.5 | J+ |
| | | D5-N-EtFOSA | 29.6 | UJ |
| | | d9-NEt-FOSE | 49.9 | UJ |
| RC23-SED-WET-0-6-01-051420 | L33094-5 | D3-N-MeFOSA | 42.7 | J+ |
| | | D5-N-EtFOSA | 34.5 | UJ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72884_3 Report date (mm/dd/yyyy): 12/2/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/23/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperatures of 7.7 and 9.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks reported in SDGs DPWG72576 and DPWG73031:<br>392602993191 (L33047-2)<br>393004067424 (L33098-1) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (sed):<br>LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | A reduced sample volume was used for sample EP18-SED-0-6-01-051220 (L33094-1) resulting in elevated reporting limits.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/23/2020

## Data Validation Qualifications for DPWG72884_3

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| RC12-SED-COMP-0-6-01-042420 | L33049-1 | 7.7 |
| RC12-SED-COMP-0-6-01-042420 | L33049-2 | 7.7 |
| RC12-SED-0-6-01-042520 | L33049-3 | 7.7 |
| EP18-SED-0-6-01-051220 | L33094-1 | 9.8 |
| GLI-SED-0-6-01-051220 | L33094-2 | 9.8 |
| RC22-SED-WET-0-6-01-051420 | L33094-3 | 9.8 |
| RC22-SED-WET-6-12-01-051420 | L33094-4 | 9.8 |
| RC23-SED-WET-0-6-01-051420 | L33094-5 | 9.8 |
| RC23-SED-WET-6-12-01-051420 | L33094-6 | 9.8 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA (CS7) | 74.8% | 75-125% | Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-SED-COMP-0-6-01-042420 | L33049-2 | D3-N-MeFOSA | 33.8 | J+ |
| | | D5-N-EtFOSA | 27.0 | J+ |
| | | d9-NEt-FOSE | 43.2 | J+ |
| CLIENT SAMPLE NO. EP18-SED-0-6-01-051220 | L33094-1 | 13C4-PFBA | 22.7 | J+ |
| | | D5-N-EtFOSA | 42.9 | UJ |
| RC22-SED-WET-0-6-01-051420 | L33094-3 | D3-N-MeFOSA | 40.5 | J+ |
| | | D5-N-EtFOSA | 29.6 | UJ |
| | | d9-NEt-FOSE | 49.9 | UJ |
| RC23-SED-WET-0-6-01-051420 | L33094-5 | D3-N-MeFOSA | 42.7 | J+ |
| | | D5-N-EtFOSA | 34.5 | UJ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72885_2 Report date (mm/dd/yyyy): 12/1/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/23/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples except for L33068-75 were received at a temperature greater than 6 °C. See Section 10 and data qualifications summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some of the field IDs were updated. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks reported in SDG DPWG73047.<br>Equipment blanks reported in DPWG73047 were all nondetect. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip Blanks:<br>392789107088 (L33068-3 R) - nondetect<br>392789107125 ( L33068-7 R) - nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | WL7-WAT-BULK-03-050720 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD; LCSD;<br>FDs: WL7-WAT-BULK-02-050720 (L33068-51 i), WL7-WAT-BULK-03-050720(L33068-52)<br>WL12-WAT-BULK-02-050720 (L33068-57 i)<br>RC21-WAT-BULK-02-050720 (L33068-61)<br>EP24-WAT-BULK-02-050720 (L33068-68) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☐ | ☒ | ☐ | 30% RPD limit for PFAS (water): |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | RC21-WAT-BULK-01-050720/RC21-WAT-BULK-02-050720:<br>PFBA: 40.9% RPD<br>See Section 10 and data qualifications summary. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/23/2020

## Data Validation Qualifications for DPWG72885_2

**Chain of custody preservation, and holding time (Section 1C):** Nine samples were received at 7.9 °C, eleven samples were received at 7.9 °C, eight samples received at 8.9 °C, and six samples received at 7.2 °C. All samples except for EP21-WAT-SML-01-042420 (L33068-75) were qualified (detects qualified as estimated biased low (J-) and nondetects UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC21-WAT-BULK-02-050720 | L33068-61 | 13C4-PFBA | 14.6 | J+ |
| EP23-WAT-BULK-01-050720 | L33068-65 | 13C4-PFBA | 25.5 | J+ |
| EP23-WAT-SML-01-050720 | L33068-66 | 13C4-PFBA | 25.2 | J+ |
| EP24-WAT-BULK-01-050720 | L33068-67 | 13C4-PFBA | 17.7 | J+ |
| EP24-WAT-BULK-02-050720 | L33068-68 | 13C4-PFBA | 25.0 | J+ |
| EP8-WAT-BULK-01-050820 | L33068-71 | 13C4-PFBA | 17.5 | J+ |
| WL18-WAT-BULK-01-050820 | L33068-72 | 13C4-PFBA | 14.9 | J+ |
| EP17-WAT-BULK-01-051120 | L33068-73 | 13C4-PFBA | 22.0 | J+ |
| EP25-WAT-BULK-01-051120 | L33068-74 | 13C4-PFBA | 14.8 | J+ |
| EP21-WAT-SML-01-042420 | L33068-75 | 13C4-PFBA | 25.9 | J+ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field precision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **RC21-WAT-BULK-01-050720/RC21-WAT-BULK-02-050720:** | | | |
| PFBA | 59.0 | 89.3 | 40.9 |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72892 Report date (mm/dd/yyyy): 7/13/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/11/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Sample EP16-WAT-BULK-01-051120 (L33071-2) received at a temperature of 7.2 °C (SDG 3000024792 contains receipt information for this sample). See Section 10 and data qualifications summary. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks reported in SDG DPWG73047:<br>392789107099 (L33068-8 R)<br>392789107066 ( L33068-2 R) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 392789107099 (L33068-8 R) - nondetect<br>392789107066 ( L33068-2 R):<br>6:2 FTS 6.92 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD;<br>FDs: EP21-FOAM-02-050520 (L33069-7) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>RPDs - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume used for all samples except EP16-WAT-BULK-01-051120 (L33071-2) due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/11/2020

## Data Validation Qualifications for DPWG72892

**Chain of custody preservation, and holding time (Section 1C):** Sample EP16-WAT-BULK-01-051120 (L33071-2) received at a temperature of 7.2 °C (SDG 3000024792 contains receipt information for this sample). Detects were qualified as estimated biased low (J-) and nondetects UJ.

**Blanks (Section 3Aii):** The 6:2 FTS result for sample RC21-FOAM-01-042320 (L33069-4) was within 10 times the trip blank concentration and qualified B.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18A-FOAM-FRESH-01-050520 | L33069-3 | 13C2-PFTeDA | 42.3 | J+ |
| | | d9-NEt-FOSE | 48.4 | UJ |
| EP21-FOAM-01-042320 | L33069-5 | 13C2-PFTeDA | 36.3 | UJ |
| EP21-FOAM-01-050520 | L33069-6 | 13C2-PFTeDA | 18.9 | UJ |
| EP21-FOAM-02-050520 | L33069-7 | 13C2-PFTeDA | 23.9 | UJ |
| EP25-FOAM-01-051120 | L33069-8 | 13C2-PFTeDA | 24.0 | UJ |
| WL11-FOAM-01-050520 | L33069-9 | 13C2-PFDoA | 49.7 | UJ |
| | | 13C2-PFTeDA | 44.1 | UJ |
| EP16-WAT-BULK-01-051120 | L33071-2 | 13C4-PFBA | 10.3 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG72962 Report date (mm/dd/yyyy): 7/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/11/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples received at temperatures of 9.0 °C and 6.5 °C. See data qualifications summary for sample list and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks reported in SDG DPWG73047:<br>392789107055 (L33068-1 R)<br>392789107099 ( L33068-8 R)<br>392789107114 (L33068-4 R)<br>392789107066 (L33068-2 R) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 392789107114 (L33068-4 R):<br>6:2 FTS 10.2 J ng/L<br>392789107066 (L33068-2 R):<br>6:2 FTS 6.92 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-050420 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MN limits 70% to 130% (water)<br>See data qualification summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - ok;<br>FDs: RC5-WAT-BULK-02-050420/ |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | RC5-WAT-BULK-03-050420 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/11/2020

## Data Validation Qualifications for DPWG72962

**Chain of custody preservation, and holding time (Section 1C):** Results for samples RC12-WAT-BULK-01-042920 (L33068-21), RC14-WAT-BULK-01-042920 (L33068-22) and RC21-WAT-BULK-01-042920 (L33068-23) were qualified (UJ and J-) due to a sample receipt temperature of 9.0 °C.

Results for samples EP16-WAT-BULK-01-042920 (L33068-24), EP19-WAT-BULK-01-042920 (L33068-25), EP20-WAT-BULK-01-042920 (L33068-26), WL6-WAT-BULK-01-042920 (L33068-27), WL9-WAT-BULK-01-042920 (L33068-28), WL12-WAT-BULK-01-042920 (L33068-29), WL15-WAT-BULK-01-042920 (L33068-30), and VB1-WAT-BULK-01-042920 (L33068-31) were qualified (UJ and J-) due to a sample receipt temperature of 6.5 °C.

**Blanks (Section 3Aii):** The 6:2 FTS results for samples EP4-WAT-BULK-01-050420 (L33068-33) and RC5-WAT-BULK-03-050420 (L33068-40 (A)) were within 10 times the trip blank concentration and qualified B.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-WAT-BULK-01-042920 | L33068-21 | 13C4-PFBA | 31.2 | J+ |
| RC14-WAT-BULK-01-042920 | L33068-22 | 13C4-PFBA | 49.3 | J+ |
| RC21-WAT-BULK-01-042920 | L33068-23 | 13C4-PFBA | 38.2 | J+ |
| EP16-WAT-BULK-01-042920 | L33068-24 | 13C4-PFBA | 33.3 | J+ |
| EP19-WAT-BULK-01-042920 | L33068-25 | 13C4-PFBA | 30.4 | J+ |
| WL6-WAT-BULK-01-042920 | L33068-27 | 13C4-PFBA | 28.7 | J+ |
| WL9-WAT-BULK-01-042920 | L33068-28 | 13C4-PFBA | 31.2 | J+ |
| WL12-WAT-BULK-01-042920 | L33068-29 | 13C4-PFBA | 29.7 | J+ |
| WL15-WAT-BULK-01-042920 | L33068-30 | 13C4-PFBA | 22.4 | J+ |
| VB1-WAT-BULK-01-042920 | L33068-31 | 13C4-PFBA | 23.8 | J+ |
| RC3-WAT-BULK-01-050420 | L33068-32 | 13C4-PFBA | 28.0 | J+ |
| EP4-WAT-BULK-01-050420 | L33068-33 | 13C4-PFBA | 11.0 | J+ |
| BP1-WAT-BULK-01-050420 | L33068-35 | 13C4-PFBA | 15.0 | J+ |
| VB1-WAT-BULK-01-050420 | L33068-36 | 13C4-PFBA | 13.0 | J+ |
| VB2-WAT-BULK-01-050420 | L33068-37 | 13C4-PFBA | 16.8 | J+ |
| VB3-WAT-BULK-01-050420 | L33068-38 | 13C4-PFBA | 21.9 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **RC5-WAT-BULK-03-050420:** | | | |
| PFOA | **184/**130 | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **--/227** | 70-130 | |
| PFHpS | **133/140** | 70-130 | The detect for sample RC5-WAT-BULK-03-050420 was qualified as estimated biased high (J+). |
| PFNS | **147/159** | 70-130 | Not detected in sample RC5-WAT-BULK-03-050420. No qualifiers. |
| PFDS | 128/**140** | 70-130 | |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73018 Report date (mm/dd/yyyy): 7/28/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/12/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Some of the samples received at temperatures of 9.4 °C and 7.2 °C. See data qualifications summary for sample list and qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | Associated trip blanks reported in SDGs DPWG73031 and DPWG73047:<br>392789107125 (L33068-7 R)<br>393004067424 ( L33098-1) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>OPR DUPLICATE (WG72616-103):<br>PFNA 67.3% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/12/2020

## Data Validation Qualifications for DPWG73018

**Chain of custody preservation, and holding time (Section 1C):** Results for sample EP16-WAT-BULK-01-051120 (L33071-2) were qualified (UJ and J-) due to a sample receipt temperature of 7.2 °C.

Results for samples EP18-WAT-BULK-01-051220 (L33098-20) were qualified (UJ and J-) due to a sample receipt temperature of 9.4 °C.

**LCS Exceedances (Section 5Bii):** PFNA detects were qualified estimated biased low (J-) and nondetects qualified UJ for samples RC5-WAT-BULK-01-050420 (L33071-1), EP16-WAT-BULK-01-051120 (L33071-2), and EP18-WAT-BULK-01-051220 (L33098-20).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73031 Report date (mm/dd/yyyy): 7/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/12/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | All samples received at elevated temperatures of 9.4 °C and 9.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks:<br>393004067424 (L33098-1)<br>393004067435 (L33098-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFOSA 0.889 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | Results qualified B by the laboratory were within 10 times the blank concentration, taking into consideration sample volumes and dilutions and the B qualifiers should be retained. The B qualifications indicate contamination and a high bias in the result. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>LCSD RPDs - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used due to high sample concentrations for samples RC23-WAT-BULK-01-051420, RC3-WAT-BULK-01-051820, RC7-WAT-BULK-01-051820, and RC20-WAT-BULK-01-051820.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/12/2020

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73031 Report date (mm/dd/yyyy): 7/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/12/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | All samples received at elevated temperatures of 9.4 °C and 9.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks:<br>393004067424 (L33098-1)<br>393004067435 (L33098-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFOSA 0.889 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | Results qualified B by the laboratory were within 10 times the blank concentration, taking into consideration sample volumes and dilutions and the B qualifiers should be retained. The B qualifications indicate contamination and a high bias in the result. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>LCSD RPDs - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used due to high sample concentrations for samples RC23-WAT-BULK-01-051420, RC3-WAT-BULK-01-051820, RC7-WAT-BULK-01-051820, and RC20-WAT-BULK-01-051820.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/12/2020

## Data Validation Qualifications for DPWG73031

**Chain of custody preservation, and holding time (Section 1C):** Results for all sample were qualified (UJ and J-) due to sample receipt temperatures of 9.4 and 9.8 °C.

**Blanks (Section 3Bii):** Results qualified B by the laboratory due to method blanks were within 10 times the blank concentration, taking into consideration sample volumes and dilutions and the B qualifiers should be retained. The B qualifications indicate contamination and a high bias in the result.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9-WAT-BULK-01-051220 | L33098-3 | 13C4-PFBA | 49.0 | J+ |
| EP15-WAT-BULK-01-051220 | L33098-4 | 13C4-PFBA | 26.1 | J+ |
| GL1-WAT-BULK-01-051220 | L33098-5 | 13C2-PFTeDA | 39.0 | UJ |
| WL6-WAT-BULK-01-051820 | L33098-8 | 13C4-PFBA | 14.2 | J+ |
| WL15-WAT-BULK-01-051820 | L33098-9 | 13C4-PFBA | 31.0 | J+ |
| VB3-WAT-BULK-01-051820 | L33098-10 | 13C4-PFBA | 9.95 | J+ |
| RC12-WAT-BULK-01-051820 | L33098-14 | 13C4-PFBA | 24.9 | J+ |
| RC14-WAT-BULK-01-051820 | L33098-15 | 13C4-PFBA | 17.5 | J+ |
| RC21-WAT-BULK-01-051820 | L33098-16 | 13C4-PFBA | 21.1 | J+ |
| EP19-WAT-BULK-01-051820 | L33098-17 | 13C4-PFBA | 15.4 | J+ |
| EP16-WAT-BULK-01-051820 | L33098-19 | 13C4-PFBA | 15.1 | J+ |
| EP18-WAT-BULK-01-051220 | L33098-20 | 13C4-PFBA | 14.4 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

**SGS AXYS DPWG73031** 3/12/21 **LS**

Initial Cal 3/18/20 **PFBA**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (13C4-PFBA) | PFBA | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | |
| | | | | | |
| 195667 | 7822 | 1.6 | 0.9994 | 0.999 | |
| 196611 | 15554 | 3.2 | 0.9889 | 0.989 | |
| 189498 | 36608 | 8 | 0.9659 | 0.966 | |
| 159457 | 151728 | 40 | 0.9515 | 0.952 | |
| 184798 | 829647 | 200 | 0.8979 | 0.898 | |
| 147235 | 3374481 | 1000 | 0.9168 | 0.917 | |
| 93880 | 8967465 | 4000 | 0.9552 | 0.955 | |
| | | Avg: | 0.9537 | 0.953655 | IS Conc = 40 |
| | | %RSD: | 3.8197 | 3.8195 | |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| EP9-WAT-BULK-01-051220 | PFBA | 761364 | 89978 | 1 | 659.69 | 660 | 0.538 | 0.004 | -3 |
| EP18-WAT-BULK-01-051220 | PFBA | 24647 | 31812 | 1 | 59.63 | 59.6 | 0.545 | 0.004 | -20 |
| LCS (WG72614-102) | PFBA | 192062 | 210362 | 1 | 38.30 | 38.3 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/30/20 2:08 | 216581 | 235798 | 38.526 | 38.5 | 40.00 | 96.25 | 96.3% |
| CCV 6/30/20 7:59 | 220518 | 239108 | 38.683 | 38.7 | 40.00 | 96.75 | 96.7% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG72614-101): | PFBA | 0.222 | <3.2 | verified <3.2 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MCPA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG73031_2 (Analysis WG72614) dated 2/22/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 3/12/2021 |

The samples listed below were collected at the Project 1007 Site on May 12 through 19, 2020.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| EP9-WAT-BULK-01-051220 | | 5/12/2020 | L33098-3 |
| EP15-WAT-BULK-01-051220 | | 5/12/2020 | L33098-4 |
| GL1-WAT-BULK-01-051220 | | 5/12/2020 | L33098-5 |
| RC22-WAT-BULK-01-051420 | | 5/14/2020 | L33098-6 |
| RC23-WAT-BULK-01-051420 | | 5/14/2020 | L33098-7 |
| WL6-WAT-BULK-01-051820 | | 5/18/2020 | L33098-8 |
| WL15-WAT-BULK-01-051820 | | 5/18/2020 | L33098-9 |
| VB3-WAT-BULK-01-051820 | | 5/18/2020 | L33098-10 |
| RC3-WAT-BULK-01-051820 | | 5/18/2020 | L33098-11 |
| RC7-WAT-BULK-01-051820 | | 5/18/2020 | L33098-12 |
| RC20-WAT-BULK-01-051820 | | 5/18/2020 | L33098-13 |
| RC12-WAT-BULK-01-051820 | | 5/18/2020 | L33098-14 |
| RC14-WAT-BULK-01-051820 | | 5/18/2020 | L33098-15 |
| RC21-WAT-BULK-01-051820 | | 5/18/2020 | L33098-16 |
| EP19-WAT-BULK-01-051820 | | 5/18/2020 | L33098-17 |
| EP20-WAT-BULK-01-051820 | | 5/18/2020 | L33098-18 |
| EP16-WAT-BULK-01-051820 | | 5/18/2020 | L33098-19 |
| EP18-WAT-BULK-01-051220 | | 5/12/2020 | L33098-20 |
| **Field QC Samples:** | | | |
| 393004067424 | Trip Blank | 5/14/2020 | L33098-1 |
| 393004067435 | Trip Blank | 5/19/2020 | L33098-2 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✗ | Laboratory and field blanks |
| ✗ | Labeled Analogs (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| NA | Field duplicates |
| ✓ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of six results that were rejected (R) due to severely low labeled analog recoveries. In addition, seven PFOSA results were qualified B due to laboratory contamination and five results were qualified J+ due to labeled analog recoveries. Data qualifications were required as listed in Table 1. Results reported below the quantitation limit were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the validator, but are not shown in Table 1.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact and above the temperature requirement of <6 °C. The samples were received at temperatures of 9.4 and 9.8 °C. The reporting limit for nondetect results were qualified as estimated (UJ) and detects were qualified as estimated biased low (J-).

Two trip blanks were received, and the sample IDs were updated to comply with MPCA chain of custody and EQuIS requirements.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90 day hold time.

## Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery). The ICALs were within the MPCA criteria, except as noted in the table below for the CS7 standard.

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration (Date) | Analyte | % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA | CS7=74.8 | 75-125 | Since the highest calibration point (CS7) was out of compliance, and results were nondetect, no qualifiers. |
| Closing CCV 6/30/20 7:59 | 9Cl-PF3ONS | 136 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 11Cl-PF3OUdS | 131 | 70-130 | |

Two CCVs were recalculated for PFBA during the validation. Results reported are verified to be correct.

## Laboratory and Field Blanks

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in method blanks, with the exception of those listed in the table below. Results within 10 times the blank concentration, taking into consideration sample prep volumes, were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Qualifiers |
|---|---|---|---|---|
| WG72614-101 | 6/26/2020 | PFOSA | 0.889 J | Results qualified B:<br><br>RC23-WAT-BULK-01-051420 (L33098-7)<br>WL15-WAT-BULK-01-051820 (L33098-9)<br>RC20-WAT-BULK-01-051820 (L33098-13)<br>RC21-WAT-BULK-01-051820 (L33098-16)<br>EP19-WAT-BULK-01-051820 (L33098-17)<br>EP20-WAT-BULK-01-051820 (L33098-18)<br>EP18-WAT-BULK-01-051220 (L33098-20) |

Two trip blanks were associated with this sample group. The trip blanks were nondetect.

## Labeled Analogs

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Six results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9-WAT-BULK-01-051220 | L33098-3 | 13C4-PFBA | 49.0 | J+ |
| EP15-WAT-BULK-01-051220 | L33098-4 | 13C4-PFBA | 26.1 | J+ |
| GL1-WAT-BULK-01-051220 | L33098-5 | 13C2-PFTeDA | 39.0 | UJ |
| WL6-WAT-BULK-01-051820 | L33098-8 | 13C4-PFBA | 14.2 | R |
| WL15-WAT-BULK-01-051820 | L33098-9 | 13C4-PFBA | 31.0 | J+ |
| VB3-WAT-BULK-01-051820 | L33098-10 | 13C4-PFBA | 9.95 | R |
| RC12-WAT-BULK-01-051820 | L33098-14 | 13C4-PFBA | 24.9 | J+ |
| RC14-WAT-BULK-01-051820 | L33098-15 | 13C4-PFBA | 17.5 | R |
| RC21-WAT-BULK-01-051820 | L33098-16 | 13C4-PFBA | 21.1 | J+ |
| EP19-WAT-BULK-01-051820 | L33098-17 | 13C4-PFBA | 15.4 | R |
| EP16-WAT-BULK-01-051820 | L33098-19 | 13C4-PFBA | 15.1 | R |
| EP18-WAT-BULK-01-051220 | L33098-20 | 13C4-PFBA | 14.4 | R |

**MS/MSD Results**

An MS/MSD was not requested for this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not included in this SDG.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFBA. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Multiple sample dilutions were performed due to high PFOS concentrations for samples RC22-WAT-BULK-01-051420 (L33098-6) and RC12-WAT-BULK-01-051820 (L33098-14).

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification.

Extraction notes indicated that the cartridge was clogged at full pressure during the prep of sample VB3-WAT-BULK-01-051820 (L33098-10), and the sample was manually processed.

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| RC23-WAT-BULK-01-051420 (L33098-7)<br>WL15-WAT-BULK-01-051820 (L33098-9)<br>RC20-WAT-BULK-01-051820 (L33098-13)<br>RC21-WAT-BULK-01-051820 (L33098-16)<br>EP19-WAT-BULK-01-051820 (L33098-17)<br>EP20-WAT-BULK-01-051820 (L33098-18)<br>EP18-WAT-BULK-01-051220 (L33098-20) | PFOSA | ng/L | B | mb |
| EP9-WAT-BULK-01-051220 (L33098-3)<br>EP15-WAT-BULK-01-051220 (L33098-4)<br>WL15-WAT-BULK-01-051820 (L33098-9)<br>RC12-WAT-BULK-01-051820 (L33098-14)<br>RC21-WAT-BULK-01-051820 (L33098-16) | PFBA | ng/L | J<br>(final qualifier J as both high and low bias present) | s |
| GL1-WAT-BULK-01-051220 (L33098-5) | PFTeDA | ng/L | UJ | s |
| WL6-WAT-BULK-01-051820 (L33098-8)<br>VB3-WAT-BULK-01-051820 (L33098-10)<br>RC14-WAT-BULK-01-051820 (L33098-15)<br>EP19-WAT-BULK-01-051820 (L33098-17)<br>EP16-WAT-BULK-01-051820 (L33098-19)<br>EP18-WAT-BULK-01-051220 (L33098-20) | PFBA | ng/L | R | s |

(1): Data Validation Qualifiers:

- B The analyte was detected in the sample at a concentration within ten times the blank concentration.
- J Estimated (+/- indicates high or low bias)
- R Rejected
- UJ Estimated reporting limit

(2): Reason Codes:

- mb Method blank
- s Surrogate (labeled analog)
- sr Sample receipt

# Attachment A

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73047 Report date (mm/dd/yyyy): 7/28/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/13/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Thirteen samples were received at elevated temperatures. See data qualifications summary for detail regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some updates were made in the Field Sample IDs. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Seven trip blanks and six equipment blanks were reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Equipment blanks were nondetect.<br>Trip blanks detects:<br>392789107066 (-2): 6:2 FTS 6.92 J ng/L<br>392789107114 (-4): 6:2 FTS 10.2 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Qualifications are discussed in checklists for DPWG72892 and DPWG72962. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water):<br>LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/13/2020

## Data Validation Qualifications for DPWG73047

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| 392789107055 | L33068-1 R | 9.0 |
| 392789107088 | L33068-3 R | 7.9 |
| 392789107103 | L33068-6 R | 8.9 |
| 392789107125 | L33068-7 R | 7.2 |
| 392789107099 | L33068-8 R | 6.5 |
| EQ-AUGER-01-051220 | L33068-13 R | 7.2 |
| EQ-PAIL-01-051220 | L33068-14 R | 7.2 |
| RC3-WAT-BULK-01-042820 | L33068-15 R | 9.0 |
| RC12-WAT-BULK-01-042820 | L33068-16 R | 9.0 |
| RC14-WAT-BULK-01-042820 | L33068-17 R | 9.0 |
| RC21-WAT-BULK-01-042820 | L33068-18 R | 9.0 |
| RC3-WAT-BULK-01-042920 | L33068-19 R | 9.0 |
| RC7-WAT-BULK-01-042920 | L33068-20 R | 9.0 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3-WAT-BULK-01-042820 | L33068-15 R | 13C4-PFBA | 41.7 | J+ |
| RC12-WAT-BULK-01-042820 | L33068-16 R | 13C4-PFBA | 7.77 | J+ |
| | | 13C5-PFPeA | 37.2 | J+ |
| | | 13C2-4:2 FTS | 47.3 | UJ |
| RC14-WAT-BULK-01-042820 | L33068-17 R | 13C4-PFBA | 4.81 | J+ |
| | | 13C5-PFPeA | 44.9 | J+ |
| RC21-WAT-BULK-01-042820 | L33068-18 R | 13C4-PFBA | 7.45 | J+ |
| | | 13C5-PFPeA | 42.5 | J+ |
| RC3-WAT-BULK-01-042920 | L33068-19 R | 13C4-PFBA | 1.73 | J+ |
| | | 13C5-PFPeA | 16.2 | J+ |
| | | 13C2-4:2 FTS | 32.1 | UJ |
| | | 13C3-HFPO-DA | 46.0 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC7-WAT-BULK-01-042920 | L33068-20 R | 13C4-PFBA | 3.30 | J+ |
| | | 13C5-PFPeA | 33.3 | J+ |
| | | 13C2-4:2 FTS | 47.6 | UJ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73050 Report date (mm/dd/yyyy): 7/29/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/7/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | The COC on page 30 was not signed by the laboratory. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples MW7A-GW-73-77-01-052620 (L33146-1) and MW7A-GW-86-90-01-052720 (L33146-2) were received at 6.2 °C. Detects were qualified estimated biased low (J-) and nondetects UJ. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV ok. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | 808335139190 (L33165-1) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank was nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See Section 10 and data qualifications summary. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW6B-GW-140-150-03-060220 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water). ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | RLs for sample MW5A-GW-HYDRA-210-220-01-060320 (L33165-3) somewhat elevated. The case narrative indicates that not all the sample passed through the SPE and this was taken into account when calculating RLs. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/7/2020

## Data Validation Qualifications for DPWG73050

**Chain of custody preservation, and holding time (Section 1C):** Samples MW7A-GW-73-77-01-052620 (L33146-1) and MW7A-GW-86-90-01-052720 (L33146-2) were received at 6.2 °C. Detects were qualified estimated biased low (J-) and nondetects were qualified UJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW3A-GW-230-250-01-060320 | L33166-2 | 13C2-PFTeDA | 48.7 | UJ |
| | | D3-N-MeFOSA | 47.7 | UJ |
| | | D5-N-EtFOSA | 46.6 | UJ |
| MW5A-GW-HYDRA-210-220-01-060320 | L33165-3 | 13C4-PFBA | 1.22 | J+ |
| | | 13C5-PFPeA | 21.5 | J+ |
| | | 13C2-PFTeDA | 45.3 | UJ |
| MW6A-GW-185-192-01-060220 | L33146-5 | 13C4-PFBA | 3.95 | J+ |
| | | 13C5-PFPeA | 42.1 | J+ |
| 808335139190 | L33165-1 | D3-N-MeFOSA | 48.4 | UJ |

**SGS AXYS DPWG73031** 3/14/21 **LS**

Initial Cal 3/18/20 **PFOS - sum**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | p 698 |
|---|---|---|---|---|---|
| IS (13C8-PFOS) | PFOS-SUM | | | | |
| | | | | | |
| 22541 | 1285 | 0.463 | 1.2313 | | |
| 19689 | 2309 | 0.952 | 1.2319 | | |
| 23818 | 5348 | 1.823 | 1.2317 | single RFs not reported | |
| 19224 | 22186 | 9.368 | 1.2319 | | |
| 22700 | 125186 | 44.767 | 1.2319 | | |
| 19429 | 542632 | 226.713 | 1.2319 | | |
| 13651 | 1707180 | 1015.1 | 1.2320 | | |
| | | Avg: | 1.2318 | 1.231880 | IS Conc = 10 |
| | | %RSD: | | 12.0465 | |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW9A-GW-96-100-01-060220 | PFOS | 163432 | 23849 | 1 | 105.16 | 105 | 0.529 | 0.004 | L33146-4 |
| MW4A-GW-140-160-01-060220 | PFOS | 223816 | 26064 | 1 | 129.33 | 129 | 0.539 | 0.004 | L33146-7 |
| 6B-GW-140-150-03-060220 (MS) | PFOS | 129339 | 21722 | 1 | 90.35 | 90.3 | 0.535 | 0.004 | WG72647-103 (MS) |
| LCS (WG72647-102) | PFOS | 26045 | 26795 | 1 | 7.89 | 7.89 | 1.00 | 0.004 | |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG72647-101): | PFOS | 0.078 | <0.800 | verified <0.800 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MCPA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG73050_2/DPWG73050_3 (Analysis WG72647) dated 2/16/21 and 3/16/21 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 3/13/2021 |

The samples listed below were collected at the Project 1007 Site on May 26 through June 8, 2020.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| MW6B-GW-140-150-03-060220 | Field Duplicate of MW6B-GW-140-150-01-060220; MS/MSD | 6/2/20 | L33145-14 (A) |
| MW7A-GW-73-77-01-052620 | | 5/26/20 | L33146-1 |
| MW7A-GW-86-90-01-052720 | | 5/27/20 | L33146-2 |
| MW9A-GW-16-20-01-060220 | | 6/2/20 | L33146-3 |
| MW9A-GW-96-100-01-060220 | | 6/2/20 | L33146-4 |
| MW6A-GW-185-192-01-060220 | | 6/2/20 | L33146-5 |
| MW6B-GW-140-150-01-060220 | | 6/2/20 | L33146-6 |
| MW4A-GW-140-160-01-060220 | | 6/2/20 | L33146-7 |
| MW5B-GW-HYDRA-110-120-01-060320 | | 6/3/20 | L33165-2 |
| MW5A-GW-HYDRA-210-220-01-060320 | | 6/3/20 | L33165-3 |
| MW3B-GW-HYDRA-110-130-01-060320 | | 6/3/20 | L33165-4 |
| MW5A-GW-210-220-01-060320 | | 6/3/20 | L33165-5 |
| MW3B-GW-110-130-01-060320 | | 6/3/20 | L33165-6 |
| MW13A-GW-286-290-01-060820 | | 6/8/20 | L33165-7 |
| MW5B-GW-110-120-01-060320 | | 6/3/20 | L33166-1 |
| MW3A-GW-230-250-01-060320 | | 6/3/20 | L33166-2 |
| MW7A-GW-200-210-01-060420 | | 6/3/20 | L33166-3 |
| **Field QC Samples:** | | | |
| 808335139190 | Trip Blank | -- | L33165-1 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

- ✓ Data completeness (chain-of-custody (COC)/sample integrity
- ✗ Sample Receipt and Holding times
- ✓ Initial calibration/continuing calibration verification
- ✓ Laboratory and field blanks
- ✗ Labeled Analogs (Surrogates)
- ✓ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
- ✓ Laboratory control sample (LCS)
- ✓ Field duplicates
- ✓ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of two results that were rejected (R) due to severely low labeled analog recoveries. In addition, results were qualified J+/UJ due to labeled analog recoveries; and J-/UJ due to sample receipt temperature. Data qualifications are listed in Table 1. Results reported below the quantitation limit were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the validator, but are not shown in Table 1.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples list below were received at the laboratory intact and above the temperature requirement of <6 °C. The reporting limit for nondetect results were qualified as estimated (UJ) and detects were qualified as estimated biased low (J-).

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| MW7A-GW-73-77-01-052620 | L33146-1 | 6.2 |
| MW7A-GW-86-90-01-052720 | L33146-2 | 6.2 |
| MW5B-GW-HYDRA-110-120-01-060320 | L33165-2 | 10.2 |

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| MW5A-GW-HYDRA-210-220-01-060320 | L33165-3 | 10.2 |
| MW3B-GW-HYDRA-110-130-01-060320 | L33165-4 | 10.2 |
| MW5A-GW-210-220-01-060320 | L33165-5 | 10.2 |
| MW3B-GW-110-130-01-060320 | L33165-6 | 10.2 |
| MW13A-GW-286-290-01-060820 | L33165-7 | 10.2 |
| MW5B-GW-110-120-01-060320 | L33166-1 | 10.2 |
| MW3A-GW-230-250-01-060320 | L33166-2 | 10.2 |
| MW7A-GW-200-210-01-060420 | L33166-3 | 10.2 |
| 808335139190 | L33165-1 | 10.2 |

One trip blank was received, and the sample ID was updated to comply with MPCA chain of custody and EQuIS requirements.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90 day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery). The ICALs were within the MPCA criteria, except as noted in the table below for the CS7 standard.

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery.

| Calibration (Date) | Analyte | % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA | CS7=74.8 | 75-125 | Since the highest calibration point (CS7) was out of compliance, and results were nondetect, no qualifiers. |

**Laboratory and Field Blanks**

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in method blanks.

One trip blank was associated with this sample group. The trip blank was nondetect.

**Labeled Analogs**

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable

MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW6A-GW-185-192-01-060220 | L33146-5 | 13C4-PFBA | 3.95 | R |
| MW6A-GW-185-192-01-060220 | L33146-5 | 13C5-PFPeA | 42.1 | J+ |
| 808335139190 | L33165-1 | D3-N-MeFOSA | 48.4 | UJ |
| MW5A-GW-HYDRA-210-220-01-060320 | L33165-3 | 13C4-PFBA | 1.22 | R |
| MW5A-GW-HYDRA-210-220-01-060320 | L33165-3 | 13C5-PFPeA | 21.5 | J+ |
| MW5A-GW-HYDRA-210-220-01-060320 | L33165-3 | 13C2-PFTeDA* | 45.3 | UJ |
| MW3A-GW-230-250-01-060320 | L33166-2 | 13C2-PFTeDA* | 48.7 | UJ |
| MW3A-GW-230-250-01-060320 | L33166-2 | D3-N-MeFOSA | 47.7 | UJ |
| MW3A-GW-230-250-01-060320 | L33166-2 | D5-N-EtFOSA | 46.6 | UJ |
| MW7A-GW-200-210-01-060420 | L33166-3 | 13C2-PFDoA | 49.0 | UJ |
| MW7A-GW-200-210-01-060420 | L33166-3 | 13C2-PFTeDA* | 42.0 | UJ |
| MW7A-GW-200-210-01-060420 | L33166-3 | D3-N-MeFOSA | 49.0 | UJ |
| MW7A-GW-200-210-01-060420 | L33166-3 | D5-N-EtFOSA | 47.9 | UJ |
| MW7A-GW-200-210-01-060420 | L33166-3 | d9-NEt-FOSE | 46.6 | UJ |

*PFTrDA also quantitated using 13C2-PFTeDA (PFTrDA also qualified).

**MS/MSD Results**

An MS/MSD was requested for sample MW6B-GW-140-150-03-060220. The MS/MSD %Recoveries (%R) and relative percent differences (RPDs)were within the MPCA limits.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Sample MW6B-GW-140-150-03-060220 (L33145-14 (A)) is considered a field duplicate of MW6B-GW-140-150-01-060220 (L33146-6). RPDs for the field duplicates were within the 50 percent limit for other matrices, or were within ± 2 times the RL.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **MW6B-GW-140-150-01-060220/ MW6B-GW-140-150-03-060220:** | | | |
| PFBA | 71.6 | 75.4 | 5.17 |
| PFPeA | 2.52 J | 2.67 J | ±RL |
| PFHxA | 3.62 | 3.48 | 3.94 |
| PFOA | 4.75 | 4.64 | 2.34 |
| PFOS | 0.889 J | 0.818 U | -- |

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. Anomalies were observed during the re-calculation and verification of the results and the laboratory indicated the results were reprocessed due to branched isomers, and resubmitted the data package. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW7A-GW-73-77-01-052620 (L33146-1)<br>MW7A-GW-86-90-01-052720 (L33146-2)<br>MW5B-GW-HYDRA-110-120-01-060320 (L33165-2)<br>MW5A-GW-HYDRA-210-220-01-060320 (L33165-3)<br>MW3B-GW-HYDRA-110-130-01-060320 (L33165-4)<br>MW5A-GW-210-220-01-060320 (L33165-5)<br>MW3B-GW-110-130-01-060320 (L33165-6)<br>MW13A-GW-286-290-01-060820 (L33165-7)<br>MW5B-GW-110-120-01-060320 (L33166-1)<br>MW3A-GW-230-250-01-060320 (L33166-2)<br>MW7A-GW-200-210-01-060420 (L33166-3)<br>808335139190 (L33165-1) | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| MW6A-GW-185-192-01-060220 (L33146-5) | PFBA | ng/L | R | s |
| | PFPeA | ng/L | J+ | s |
| 808335139190 (L33165-1) | MeFOS | ng/L | UJ | s |
| MW5A-GW-HYDRA-210-220-01-060320 (L33165-3) | PFBA | ng/L | R | s |
| | PFPeA | ng/L | J+ | s |
| | PFTeDA<br>PFTrDA | ng/L | UJ | s |
| MW3A-GW-230-250-01-060320 (L33166-2) | PFTeDA<br>PFTrDA<br>N-MeFOSA<br>N-EtFOSA | ng/L | UJ | s |
| MW7A-GW-200-210-01-060420 (L33166-3) | PFDoA<br>PFTeDA<br>PFTrDA<br>N-MeFOSA<br>N-EtFOSA<br>N-Et-FOSE | ng/L | UJ | s |

(1): Data Validation Qualifiers:

| | |
|---|---|
| J | Estimated (+/- indicates high or low bias) |
| R | Rejected |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| s | Surrogate (labeled analog) |
| sr | Sample receipt |

**Attachment A**

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73327_2 Report date (mm/dd/yyyy): 12/1/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/26/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at a temperature greater than 6 °C. See data qualifications summary for exceedances and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some of the field IDs were updated (trip blanks). |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Three trip blanks reported:<br>808335139156 (L33267-1)<br>808335139145 (L33267-2)<br>808335139167 (L33267-3)<br>Equipment blank:<br>EQ-DRILLERPUMP-062420 (L33267-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 808335139167 (L33267-3):<br>6:2 FTS 3.49 J ng/L<br>EQ-DRILLERPUMP-062420 (L33267-4):<br>PFOS 3.24 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Four results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD;<br>FDs: MW2A-GW-36-40-01-062220/MW2A- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | GW-36-40-02-062220 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit for PFAS (water):<br>See Section 10 and data qualifications summary. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Lower sample volumes used for samples GL2-FOAM-01-062920 (L33266-1) and FC2-FOAM-01-062920 (L33266-2) due to high sample concentrations.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Four results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/26/2020

## Data Validation Qualifications for DPWG73327_2

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| GL2-FOAM-01-062920 | L33266-1 | 7.1 |
| FC2-FOAM-01-062920 | L33266-2 | 7.1 |
| 808335139156 | L33267-1 | 13.4 |
| 808335139145 | L33267-2 | 9.5 |
| 808335139167 | L33267-3 | 7.1 |
| EQ-DRILLERPUMP-062420 | L33267-4 | Not Listed |
| MW2A-GW-11-12-01-062220 | L33267-5 | 9.5 |
| MW2A-GW-36-40-01-062220 | L33267-6 | 9.5 |
| MW2A-GW-36-40-02-062220 | L33267-7 | 9.5 |
| MW2A-GW-66-70-01-062220 | L33267-8 | 9.5 |
| MW2A-GW-86-90-01-062220 | L33267-9 | 9.5 |
| MW2A-GW-116-120-01-062320 | L33267-10 | 9.5 |
| MW2A-GW-136-140-01-062320 | L33267-11 | 9.5 |
| MW2A-GW-206-210-01-062320 | L33267-12 | 9.5 |
| RC3-WAT-BULK-01-062920 | L33267-13 | 13.4 |
| RC7-WAT-BULK-01-062920 | L33267-14 | 13.4 |
| RC12-WAT-BULK-01-062920 | L33267-15 | 13.4 |
| RC14-WAT-BULK-01-062920 | L33267-16 | 13.4 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA (CS7) | 74.8% | 75-125% | Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |

**Field QC Blank detections (Section 3Aii):** Detects within 10 times the trip blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| 808335139167 (L33267-3) | 6:2 FTS | 3.49 J | MW2A-GW-11-12-01-062220 (L33267-5)<br>MW2A-GW-66-70-01-062220 (L33267-8) |
| EQ-DRILLERPUMP-062420 (L33267-4) | PFOS | 3.24 | RC14-WAT-BULK-01-062920 (L33267-16) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as

estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| GL2-FOAM-01-062920 | L33266-1 | 13C2-PFDoA | 43.1 | J+ |
| GL2-FOAM-01-062920 | L33266-1 | 13C2-PFTeDA | 24.8 | UJ |
| FC2-FOAM-01-062920 | L33266-2 | 13C7-PFUnA | 49.6 | J+ |
| FC2-FOAM-01-062920 | L33266-2 | 13C2-PFDoA | 45.3 | J+ |
| FC2-FOAM-01-062920 | L33266-2 | 13C2-PFTeDA | 45.1 | J+ |
| 808335139167 | L33267-3 | 13C4-PFBA | 31.9 | UJ |
| 808335139167 | L33267-3 | 13C5-PFPeA | 33.3 | UJ |
| 808335139167 | L33267-3 | 13C5-PFHxA | 34.2 | UJ |
| 808335139167 | L33267-3 | 13C4-PFHpA | 34.5 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOA | 31.9 | UJ |
| 808335139167 | L33267-3 | 13C9-PFNA | 32.6 | UJ |
| 808335139167 | L33267-3 | 13C6-PFDA | 36.5 | UJ |
| 808335139167 | L33267-3 | 13C7-PFUnA | 36.3 | UJ |
| 808335139167 | L33267-3 | 13C2-PFDoA | 40.4 | UJ |
| 808335139167 | L33267-3 | 13C2-PFTeDA | 38.9 | UJ |
| 808335139167 | L33267-3 | 13C3-PFBS | 39.6 | UJ |
| 808335139167 | L33267-3 | 13C3-PFHxS | 38.2 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOS | 41.5 | UJ |
| 808335139167 | L33267-3 | 13C2-4:2 FTS | 29.9 | UJ |
| 808335139167 | L33267-3 | 13C2-6:2 FTS | 23.4 | UJ |
| 808335139167 | L33267-3 | 13C2-8:2 FTS | 31.4 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOSA | 29.2 | UJ |
| 808335139167 | L33267-3 | D3-N-MeFOSA | 23.5 | UJ |
| 808335139167 | L33267-3 | D5-N-EtFOSA | 26.9 | UJ |
| 808335139167 | L33267-3 | D3-MeFOSAA | 27.2 | UJ |
| 808335139167 | L33267-3 | D5-EtFOSAA | 27.3 | UJ |
| 808335139167 | L33267-3 | d7-NMe-FOSE | 30.6 | UJ |
| 808335139167 | L33267-3 | d9-NEt-FOSE | 33.7 | UJ |
| 808335139167 | L33267-3 | 13C3-HFPO-DA | 36 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | 13C2-PFDoA | 43.1 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2A-GW-66-70-01-062220 | L33267-8 | 13C2-PFTeDA | 24.8 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | D3-N-MeFOSA | 23.5 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | D5-N-EtFOSA | 26.9 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | d7-NMe-FOSE | 30.6 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | d9-NEt-FOSE | 33.7 | UJ |
| RC3-WAT-BULK-01-062920 | L33267-13 | 13C4-PFBA | 17.9 | R |
| RC7-WAT-BULK-01-062920 | L33267-14 | 13C4-PFBA | 3.14 | R |
| RC7-WAT-BULK-01-062920 | L33267-14 | 13C5-PFPeA | 26.4 | J+ |
| RC12-WAT-BULK-01-062920 | L33267-15 | 13C4-PFBA | 6.04 | R |
| RC14-WAT-BULK-01-062920 | L33267-16 | 13C4-PFBA | 3.15 | R |
| RC14-WAT-BULK-01-062920 | L33267-16 | 13C5-PFPeA | 25.1 | J+ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field precision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **MW2A-GW-36-40-01-062220/MW2A-GW-36-40-02-062220:** | | | |
| PFBA | 50 | 85.7 | **52.6** |
| PFPeA | 9.47 | 14.9 | **44.6** |
| PFBS | 4.45 | 6.34 | **35.0** |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

**SGS AXYS DPWG73327_3** 3/21/21 **LS**

Initial Cal 3/18/20 **PFBA**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (13C4-PFBA) | PFBA | | | |
| 195667 | 7822 | 1.6 | 0.9994 | 0.999 |
| 196611 | 15554 | 3.2 | 0.9889 | 0.989 |
| 189498 | 36608 | 8 | 0.9659 | 0.966 |
| 159457 | 151728 | 40 | 0.9515 | 0.952 |
| 184798 | 829647 | 200 | 0.8979 | 0.898 |
| 147235 | 3374481 | 1000 | 0.9168 | 0.917 |
| 93880 | 8967465 | 4000 | 0.9552 | 0.955 |
| | | Avg: | 0.9537 | 0.953655 |
| | | %RSD: | 3.8197 | 3.8195 |

IS Conc = 40

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW2A-GW-11-12-01-062220 | PFBA | 1379591 | 195855 | 1 | 554 | 554 | 0.533 | 0.004 | L33267-5 |
| MW2A-GW-116-120-01-062320 | PFBA | 822783 | 190331 | 1 | 342 | 342 | 0.530 | 0.004 | L33267-10 |
| LCS (WG72614-102) | PFBA | 122195 | 240618 | 1 | 21.30 | 21.3 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 7/23/20 20:04 | 160854 | 169587 | 39.784 | 39.8 | 40.00 | 99.5 | 99.5% |
| CCV 7/27/20 20:19 | 143622 | 154903 | 38.889 | 38.9 | 40.00 | 97.25 | 97.2% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG72614-101): | PFBA | 1.148 | <3.2 | verified <3.2 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MCPA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG73327_3 (Analysis WG72857) dated 2/16/2021 | |
| Analyses/Method: | SGS AXYS Method MLA-110 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 3/21/2021 |

The samples listed below were collected at the Project 1007 Site on June 22 through 30, 2020.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| GL2-FOAM-01-062920 | | 6/29/20 | L33266-1 |
| FC2-FOAM-01-062920 | | 6/29/20 | L33266-2 |
| MW2A-GW-11-12-01-062220 | | 6/22/20 | L33267-5 |
| MW2A-GW-36-40-01-062220 | | 6/22/20 | L33267-6 |
| MW2A-GW-36-40-02-062220 | Field Duplicate of MW2A-GW-36-40-01-062220 | 6/22/20 | L33267-7 |
| MW2A-GW-66-70-01-062220 | | 6/22/20 | L33267-8 |
| MW2A-GW-86-90-01-062220 | | 6/22/20 | L33267-9 |
| MW2A-GW-116-120-01-062320 | | 6/23/20 | L33267-10 |
| MW2A-GW-136-140-01-062320 | | 6/23/20 | L33267-11 |
| MW2A-GW-206-210-01-062320 | | 6/23/20 | L33267-12 |
| RC3-WAT-BULK-01-062920 | | 6/29/20 | L33267-13 |
| RC7-WAT-BULK-01-062920 | | 6/29/20 | L33267-14 |
| RC12-WAT-BULK-01-062920 | | 6/29/20 | L33267-15 |
| RC14-WAT-BULK-01-062920 | | 6/29/20 | L33267-16 |
| **Field QC Samples:** | | | |
| EQ-DRILLERPUMP-062420 | Equipment Blank | 6/24/20 | L33267-4 |
| 808335139156 | Trip Blank | 6/29/20 | L33267-1 |
| 808335139145 | Trip Blank | 6/24/20 | L33267-2 |
| 808335139167 | Trip Blank | 6/30/20 | L33267-3 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and field blanks |
| ✗ | Labeled Analogs (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| ✗ | Field duplicates |
| ✓ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of four results that were rejected (R) due to severely low labeled analog recoveries. In addition, results were qualified estimated due to labeled analog recoveries, sample receipt temperature, and field duplicate imprecision. Data qualifications are listed in Table 1. Results reported below the quantitation limit were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the validator, but are not shown in Table 1.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact and above the temperature requirement of <6 °C. The sample receipt temperatures ranged from 7.1 to 13.4 °C. The reporting limit for nondetect results were qualified as estimated (UJ) and detects were qualified as estimated biased low (J-).

Three trip blanks were reported in this SDG, and the sample IDs were updated to comply with MPCA chain of custody and EQuIS requirements.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90 day hold time.

## Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery). The ICALs were within the MPCA criteria, except as noted in the table below for the CS7 standard.

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery.

| Calibration (Date) | Analyte | % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA | CS7=74.8 | 75-125 | Since the highest calibration point (CS7) was out of compliance, and results were nondetect or detected below the quantitation limit, no qualifiers. |

Two CCVs were recalculated for PFBA during the validation. Results reported are verified to be correct.

## Laboratory and Field Blanks

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in method blanks.

Three trip blanks and one equipment blank were reported in this sample group. The field QC blanks were nondetect, except as listed below.

| Blank ID | Sample Date | Compound | Blank Concentration (ng/L) | Qualifiers |
|---|---|---|---|---|
| 808335139167 (L33267-3) | 6/30/20 | 6:2 FTS | 3.49 J | Associated sample results (L33266-1 and L33266-2) were nondetect. No qualifiers. |
| EQ-DRILLERPUMP-062420 (L33267-4) | 6/24/20 | PFOS | 3.24 | Associated sample concentrations were nondetect, or greater than 10 times the blank concentration. No qualifiers. |

## Labeled Analogs

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| GL2-FOAM-01-062920 | L33266-1 | 13C2-PFDoA | 43.1 | J+ |
| GL2-FOAM-01-062920 | L33266-1 | 13C2-PFTeDA[1] | 24.8 | J+/UJ |
| FC2-FOAM-01-062920 | L33266-2 | 13C7-PFUnA | 49.6 | J+ |
| FC2-FOAM-01-062920 | L33266-2 | 13C2-PFDoA | 45.3 | J+ |
| FC2-FOAM-01-062920 | L33266-2 | 13C2-PFTeDA[1] | 45.1 | J+ |
| 808335139167 | L33267-3 | 13C4-PFBA | 31.9 | UJ |
| 808335139167 | L33267-3 | 13C5-PFPeA | 33.3 | UJ |
| 808335139167 | L33267-3 | 13C5-PFHxA | 34.2 | UJ |
| 808335139167 | L33267-3 | 3C4-PFHpA | 35.4 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOA | 31.9 | UJ |
| 808335139167 | L33267-3 | 13C9-PFNA | 32.6 | UJ |
| 808335139167 | L33267-3 | 3C6-PFDA | 36.5 | UJ |
| 808335139167 | L33267-3 | 13C7-PFUnA | 36.3 | UJ |
| 808335139167 | L33267-3 | 13C2-PFDoA | 40.4 | UJ |
| 808335139167 | L33267-3 | 13C2-PFTeDA[1] | 38.9 | UJ |
| 808335139167 | L33267-3 | 13C3-PFBS | 39.6 | UJ |
| 808335139167 | L33267-3 | 13C3-PFHxS[2] | 38.2 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOS[3] | 41.5 | UJ |
| 808335139167 | L33267-3 | 13C2-4:2 FTS | 29.9 | UJ |
| 808335139167 | L33267-3 | 13C2-6:2 FTS | 23.4 | J+ |
| 808335139167 | L33267-3 | 13C2-8:2 FTS | 31.4 | UJ |
| 808335139167 | L33267-3 | 13C8-PFOSA | 29.2 | UJ |
| 808335139167 | L33267-3 | D3-N-MeFOSA | 23.5 | UJ |
| 808335139167 | L33267-3 | D5-N-EtFOSA | 26.9 | UJ |
| 808335139167 | L33267-3 | D3-MeFOSAA | 27.2 | UJ |
| 808335139167 | L33267-3 | D5-EtFOSAA | 27.3 | UJ |
| 808335139167 | L33267-3 | d7-NMe-FOSE | 30.6 | UJ |
| 808335139167 | L33267-3 | d9-NEt-FOSE | 33.7 | UJ |
| 808335139167 | L33267-3 | 13C3-HFPO-DA[4] | 36.0 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | 13C2-PFDoA | 40.8 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | 13C2-PFTeDA[1] | 20.0 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | D3-N-MeFOSA | 41.3 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | D5-N-EtFOSA | 35.4 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | d7-NMe-FOSE | 45.3 | UJ |
| MW2A-GW-66-70-01-062220 | L33267-8 | d9-NEt-FOSE | 36.6 | UJ |
| RC3-WAT-BULK-01-062920 | L33267-13 | 13C4-PFBA | 17.9 | R |
| RC7-WAT-BULK-01-062920 | L33267-14 | 13C4-PFBA | 3.14 | R |
| RC7-WAT-BULK-01-062920 | L33267-14 | 13C5-PFPeA | 26.4 | J+ |
| RC12-WAT-BULK-01-062920 | L33267-15 | 13C4-PFBA | 6.04 | R |
| RC14-WAT-BULK-01-062920 | L33267-16 | 13C4-PFBA | 3.15 | R |
| RC14-WAT-BULK-01-062920 | L33267-16 | 13C5-PFPeA | 25.1 | J+ |

[1] PFTrDA also quantitated using 13C2-PFTeDA (PFTrDA also qualified).
[2] PFPeS also quantitated using 13C3-PFHxS (PFPeS also qualified).
[3] PFHpS, PFNS, PFDS, and PFDoS also quantitated using 13C8-PFOS (PFHpS, PFNS, PFDS, and PFDoS also qualified).
[4] ADONA, 9Cl-PF3ONS, and 11Cl-PF3OUdS also quantitated using 13C3-HFPO-DA (ADONA, 9Cl-PF3ONS, and 11Cl-PF3OUdS also qualified).

**MS/MSD Results**

An MS/MSD was not requested for this data set.

## LCS Results

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits.

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Sample MW2A-GW-36-40-02-062220 (L33267-7) was collected as a field duplicate of MW2A-GW-36-40-01-062220 (L33267-6). RPDs for the field duplicates were within the 30 percent limit for the groundwater matrix, or were within ± the RL, except as indicated in bold. Results associated with field imprecision were qualified as estimated (J).

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **MW2A-GW-36-40-01-062220/MW2A-GW-36-40-02-062220:** | | | |
| PFBA | 50 | 85.7 | **52.6** |
| PFPeA | 9.47 | 14.9 | **44.6** |
| PFHxA | 13.5 | 17.4 | 25.2 |
| PFHpA | 10.3 | 11.9 | 14.4 |
| PFOA | 102 | 111 | 8.45 |
| PFNA | 2.66 | 2.8 | 5.13 |
| PFDA | 5.78 | 5.61 | 2.99 |
| PFBS | 4.45 | 6.34 | **35.0** |
| PFPeS | 4.18 | 4.78 | 13.4 |
| PFHxS | 12.4 | 13 | 4.72 |
| PFHpS | 5.37 | 5.3 | 1.31 |
| PFOS | 718 | 702 | 2.25 |
| PFOSA | 1.55 J | 1.49 J | 3.90 |

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFBA for two samples and the LCS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| FC2-FOAM-01-062920 (L33266-2) | PFDoA<br>PFTrDA | ng/L | J+ | s |
| | PFTeDA | ng/L | UJ | s |
| FC2-FOAM-01-062920 (L33266-2) | PFUnA<br>PFDoA<br>PFTeDA<br>PFTrDA | ng/L | J+ | s |
| 808335139167 (L33267-3) | All PFAS Analytes | ng/L | Detects: J+<br>Nondetect: UJ | s |
| MW2A-GW-66-70-01-062220 (L33267-8) | PFDoA<br>PFTeDA<br>PFTrDA<br>N-MeFOSA<br>N-EtFOSA<br>N-Me-FOSE<br>N-Et-FOSE | ng/L | UJ | s |
| RC3-WAT-BULK-01-062920 (L33267-13)<br>RC12-WAT-BULK-01-062920 (L33267-15) | PFBA | ng/L | R | s |
| RC7-WAT-BULK-01-062920 (L33267-14)<br>RC14-WAT-BULK-01-062920 (L33267-16) | PFBA | ng/L | R | s |
| | PFPeA | ng/L | J+ | s |
| MW2A-GW-36-40-01-062220 (L33267-6)<br>MW2A-GW-36-40-02-062220 (L33267-7) | PFBA<br>PFPeA<br>PFBS | ng/L | J | fd |

(1): Data Validation Qualifiers:

- J Estimated (+/- indicates high or low bias)
- R Rejected
- UJ Estimated reporting limit

(2): Reason Codes:

- fd Field duplicate precision
- s Surrogate (labeled analog)
- sr Sample receipt

# Attachment A

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73328 Report date (mm/dd/yyyy): 8/28/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Five samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV<br>CCV 7/9/2020 (2:24) 8:2 FTS 151%<br>This analyte was not reported for this SDG |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks reported in EDD V72646PFAS:<br>393509322683 (L33145-1)<br>393509053158 (L33145-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | 30% limit for PFAS (water):<br>PFNS 30.6%<br>Associated results were nondetect, and were |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | acceptable without qualification. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume used (approximately 0.06 L as compared to 0.5 L). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/13/2020

## Data Validation Qualifications for DPWG73328

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| MW7A-GW-73-77-01-052620 | L33146-1 | 6.2 |
| MW7A-GW-86-90-01-052720 | L33146-2 | 6.2 |
| MW5B-GW-110-120-01-060320 | L33166-1 | 10.2 |
| MW3A-GW-230-250-01-060320 | L33166-2 | 10.2 |
| MW7A-GW-200-210-01-060420 | L33166-3 | 10.2 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW7A-GW-73-77-01-052620 | L33146-1 | 13C4-PFBA | 43.9 | J+ |
| MW6B-GW-140-150-01-060220 | L33146-6 | 13C4-PFBA | 9.93 | J+ |
| MW4A-GW-140-160-01-060220 | L33146-7 | 13C4-PFBA | 35.4 | J+ |
| MW5B-GW-110-120-01-060320 | L33166-1 | 13C4-PFBA | 48.8 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73351_2 Report date (mm/dd/yyyy): 12/2/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/26/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at a temperature greater than 6 °C. See data qualifications summary for exceedances and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some of the field IDs were updated (trip blanks). |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Two trip blanks associated (reported in SDG DPWG73327_2):<br>808335139156 (L33267-1)<br>808335139145 (L33267-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Associated trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Eight results were rejected (R) due to severely low labeled |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit for PFAS (water): |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Eight results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/26/2020

## Data Validation Qualifications for DPWG73351_2

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| RC20-WAT-BULK-01-062920 | L33267-17 | 13.4 |
| RC21-WAT-BULK-01-062920 | L33267-18 | 13.4 |
| EP18-WAT-BULK-01-062920 | L33267-19 | 13.4 |
| EP19-WAT-BULK-01-062920 | L33267-20 | 13.4 |
| EP20-WAT-BULK-01-062920 | L33267-21 | 13.4 |
| GL2-WAT-BULK-01-062920 | L33267-22 | 13.4 |
| EP16-WAT-BULK-01-062920 | L33267-23 | 7.1 |
| FC2-WAT-BULK-01-062920 | L33267-24 | 7.1 |
| WL6-WAT-BULK-01-062920 | L33267-25 | 7.1 |
| WL15-WAT-BULK-01-062920 | L33267-26 | 7.1 |
| VB3-WAT-BULK-01-062920 | L33267-27 | 7.1 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| ICAL 3/18/20 | PFTeDA (CS7) | 74.8% | 75-125% | Results were nondetects and qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Eight results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC20-WAT-BULK-01-062920 | L33267-17 | 13C4-PFBA | 8.49 | R |
| RC21-WAT-BULK-01-062920 | L33267-18 | 13C4-PFBA | 22.1 | J+ |
| EP18-WAT-BULK-01-062920 | L33267-19 | 13C4-PFBA | 18.6 | R |
| EP19-WAT-BULK-01-062920 | L33267-20 | 13C4-PFBA | 46.2 | J+ |
| EP20-WAT-BULK-01-062920 | L33267-21 | 13C4-PFBA | 34.1 | J+ |
| GL2-WAT-BULK-01-062920 | L33267-22 | 13C4-PFBA | 10.8 | R |
| GL2-WAT-BULK-01-062920 | L33267-22 | 13C2-PFTeDA | 48.7 | UJ |
| EP16-WAT-BULK-01-062920 | L33267-23 | 13C4-PFBA | 7.07 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FC2-WAT-BULK-01-062920 | L33267-24 | 13C4-PFBA | 5.01 | R |
| FC2-WAT-BULK-01-062920 | L33267-24 | 13C5-PFPeA | 46.4 | J+ |
| FC2-WAT-BULK-01-062920 | L33267-24 | 13C2-PFTeDA | 46.6 | UJ |
| WL6-WAT-BULK-01-062920 | L33267-25 | 13C4-PFBA | 8.84 | R |
| WL6-WAT-BULK-01-062920 | L33267-25 | 13C2-PFTeDA | 43.1 | UJ |
| WL15-WAT-BULK-01-062920 | L33267-26 | 13C4-PFBA | 12.9 | R |
| VB3-WAT-BULK-01-062920 | L33267-27 | 13C4-PFBA | 7.66 | R |
| VB3-WAT-BULK-01-062920 | L33267-27 | 13C2-PFTeDA | 36.2 | UJ |
| VB3-WAT-BULK-01-062920 | L33267-27 | D5-N-EtFOSA | 45.4 | UJ |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73358 Report date (mm/dd/yyyy): 8/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>ICAL: PFTeDA CS7 74.8% (limits 75% to 125%)<br>Results previoulsy qualified due to receipt temperatures, no futher qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks reported in EDD V72776:<br>808335139134 (L33230-1)<br>808335139178 (L33230-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Associated trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW13B-SOIL-COMP-91-95-03-061520 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD; FDs: MW13B-SOIL-COMP-91-95-01-061520/MW13B-SOIL-COMP-91-95-02-061520/MW13B-SOIL-COMP-91-95-03-061520<br>MW13B-SOIL-COMP-221-225-01-061620/MW13B-SOIL-COMP-221-225-02-061620 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% limit for PFAS (soils):<br>MSD - ok<br>See data qualifications summary for field duplicate qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/14/2020

## Data Validation Qualifications for DPWG73358

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| MW13B-SOIL-11-14-01-061520 | L33233-1 | 9.3 |
| MW13B-SOIL-20-22-01-061520 | L33233-2 | 9.3 |
| MW13B-SOIL-22-24-01-061520 | L33233-3 | 9.3 |
| MW13B-SOIL-COMP-91-95-01-061520 | L33233-4 N | 9.3 |
| MW13B-SOIL-COMP-91-95-02-061520 | L33233-5 N | 9.3 |
| MW13B-SOIL-COMP-91-95-03-061520 | L33233-6 (A) | 9.3 |
| MW13B-SOIL-COMP-181-185-01-061620 | L33233-7 | 10.6 |
| MW13B-SOIL-COMP-221-225-01-061620 | L33233-8 N | 10.6 |
| MW13B-SOIL-COMP-221-225-02-061620 | L33233-9 | 10.6 |
| MW13B-SOIL-COMP-266-270-01-061620 | L33233-10 | 10.6 |
| MW2A-SOIL-8-10-01-062220 | L33233-11 | 10.6 |
| MW2A-SOIL-COMP-36-40-01-062220 | L33233-12 | 10.6 |
| MW2A-SOIL-COMP-66-70-01-062220 | L33233-13 | 10.6 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13B-SOIL-20-22-01-061520 | L33233-2 | D5-N-EtFOSA | 49.2 | UJ |
| | | d7-NMe-FOSE | 49.2 | UJ |
| MW13B-SOIL-COMP-181-185-01-061620 | L33233-7 | D3-N-MeFOSA | 49.4 | UJ |
| | | d7-NMe-FOSE | 46.3 | UJ |
| | | d9-NEt-FOSE | 49.7 | UJ |
| MW13B-SOIL-COMP-221-225-01-061620 | L33233-8 N | D3-N-MeFOSA | 43.2 | UJ |
| | | D5-N-EtFOSA | 42.4 | UJ |
| | | d7-NMe-FOSE | 48.4 | UJ |
| MW2A-SOIL-COMP-66-70-01-062220 | L33233-13 | D5-N-EtFOSA | 49.9 | UJ |

**Field Duplicate Precision (6Bi):**

Results qualified as estimated (J) due to field precision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Triplicate Result (ng/g) | RPD (%) |
|---|---|---|---|---|
| **MW13B-SOIL-COMP-91-95-01-061520/MW13B-SOIL-COMP-91-95-02-061520/ MW13B-SOIL-COMP-91-95-03-061520:** | | | | |
| 6:2 FTS | 1.82 J | 0.284 U | 3.46 | >±2xRL |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73364 Report date (mm/dd/yyyy): 8/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed:<br>ICAL: PFTeDA CS7 74.8% (limits 75 to 125%)<br>Results previoulsy qualified due to receipt temperatures, no futher qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks reported in EDD V72776:<br>808335139134 (L33230-1)<br>808335139178 (L33230-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Associated trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 0.436 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | Associated samples were nondetect. |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDF |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 50% limit for PFAS (soils)<br>LSCD - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/14/2020

## Data Validation Qualifications for DPWG73364

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| MW13B-SOIL-COMP-46-50-01-061520 | L33232-1 | 9.3 |
| MW13B-SOIL-COMP-121-125-01-061520 | L33232-2 | 9.3 |
| MW2A-SOIL-11-12-01-062220 | L33232-3 | 10.6 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73383 Report date (mm/dd/yyyy): 8/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/14/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV:<br>ICAL: PFTeDA CS7 74.8% (limits 75 to 125%)<br>See data qualifications summary for qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | . |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (waters)<br>LSCD:<br>PFOSA 34.4%<br>See data qualifications summary for qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume used for sample EP18-FOAM-01-070620 (L33318-2) due to high concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**Containers for sample EP18-WAT-BULK-01-070620 (L33318-1) cracked during storage due to freezing. The sample was transferred to new containers.**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/14/2020

## Data Validation Qualifications for DPWG73383

**Calibration (Section 2A):** PFTeDA results were nondetect and qualified as estimated (UJ) for samples EP18-WAT-BULK-01-070620 (L33318-1) and EP18-FOAM-01-070620 (L33318-2).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ):

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18-FOAM-01-070620 | L33318-2 | 13C2-PFTeDA | 43.6 | UJ |

**LCSD Relative Percent Differences (RPDs) (Section 6Bi):** The PFOSA detect for sample EP18-FOAM-01-070620 (L33318-2) was qualified as estimated (J) due to a high LCS/LCSD RPD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73661 Report date (mm/dd/yyyy): 9/22/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/18/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip blanks reported in EDDs V72776 and V72857:<br>808335139134 (L33230-1)<br>808335139178 (L33230-2)<br>808335139145 (L33267-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Associated trip blanks were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | . |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (waters) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume used due to high concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/18/2020

## Data Validation Qualifications for DPWG73661

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| MW13B-GW-46-50-01-061520 | L33231-1 | 9.3 |
| MW13B-GW-121-125-01-061520 | L33231-2 | 10.6 |
| MW2A-GW-36-40-01-062220 | L33267-6 | 9.5 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13B-GW-121-125-01-061520 | L33231-2 | 13C3-PFBA | 37.4 | J+ |

**LCSD Recoveries (Section 5Bii):** LCSD recovery exceedances and qualifications are listed below:

| Analyte | % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| PFNA | 58.5 | 70-130 | The associated results were nondetect and qualified UJ:<br><br>MW13B-GW-46-50-01-061520 (L33231-1)<br>MW13B-GW-121-125-01-061520 (L33231-2)<br>MW2A-GW-36-40-01-062220 (L33267-6) |
| PFDoS | 65.2 | 70-130 | |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73712 Report date (mm/dd/yyyy): 9/22/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/18/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | All samples were received at an elevated temperature of 8.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | The sample ID for the trip blank was updated. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank:<br>809964326003 (L33383-1)<br>Equipment Blank:<br>EQ-PIPE-01-072420 (L33383-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Trip Blank detects:<br>PFOSA 1.11 J ng/L<br>The equipment blank was nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | . |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | 30% limit for PFAS (waters):<br>PFNS 30.3%<br>See data qualifications summary for details |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume used due to high concentrations for sample RC21-FOAM-01-072220 (L33383-8).<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☐ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/19/2020

## Data Validation Qualifications for DPWG73712

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at 8.9 °C and results were qualified (UJ and J-) due to sample receipt temperatures.

**Trip Blank detections (Section 3Aii):** The PFOSA result for sample EP17-WAT-BULK-01-072820 (L33383-7) was within 10 times the trip blank concentration and was qualified B.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-WAT-BULK-01-072820 | L33383-5 | 13C4-PFBA | 29.1 | J+ |
| RC21-FOAM-01-072220 | L33383-8 | 13C2-PFTeDA | 48.1 | J+ |
| MW13B-GW-295-305-01-072320 | L33384-1 | 13C2-4:2 FTS | 217 | No qualifiers |

**LCSD RPDs (Section 6Bi):** LCSD RPD exceedances and qualifications are listed below:

| Analyte | % RPD | RPD Limits | Qualifiers |
|---|---|---|---|
| PFNS | 30.3 | 30 | The associated detects were qualified as estimated (J):<br><br>RC12-WAT-BULK-01-072820 (L33383-5)<br>RC21-FOAM-01-072220 (L33383-8) |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73897 Report date (mm/dd/yyyy): 10/28/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/18/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Four samples were received at an elevated temperature of 6.5 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The sample IDs for the trip blanks were updated. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks:<br>8103 4951 0235 (L33405-1)<br>8083 3513 9237 (L33405-2)<br>8149 0205 4624 (L33405-3)<br>8083 3513 9226 (L33405-4)<br>Equipment Blanks:<br>EQ-PAIL-073120 (L33405-5)<br>EQ-DIPPER-073120 (L33405-6) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☐ | Trip Blank detects:<br>8083 3513 9237 (L33405-2):<br>6:2 FTS 1.65 J ng/L<br>8149 0205 4624 (L33405-3):<br>PFOS 0.659 J ng/L<br>8083 3513 9226 (L33405-4):<br>PFOSA 0.458 J ng/L<br>The equipment blanks were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | . |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC7-WAT-BULK-03-072820 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD, FDs:<br>RC7-WAT-BULK-01-072820/RC7-WAT-BULK-02-072820/RC7-WAT-BULK-03-072820<br>(Parent and 03 reported in SDG DPWG74066): WL9-WAT-BULK-01-073120/WL9-WAT-BULK-02-073120/WL9-WAT-BULK-03-073120<br>Parent reported in SDG DPWG74066: EP19-WAT-BULK-01-072920/EP19-WAT-BULK-02-072920 |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% limit for PFAS (waters):<br>MSD RPDs ok<br>FD - See data qualifications summary for details and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/20/2020

## Data Validation Qualifications for DPWG73897

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| 8149 0205 4624 | L33405-3 | 6.5 |
| RC16A-WAT-BULK-01-072920 | L33405-17 | 6.5 |
| RC24-WAT-BULK-01-072920 | L33405-18 | 6.5 |
| EP19-WAT-BULK-02-072920 | L33405-19 | 6.5 |

**Trip Blank detections (Section 3Aii):** Detects within 10 times the trip blank concentrations were qualified B.

| Trip Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| 8083 3513 9237 (L33405-2) | 6:2 FTS | 1.65 J | Associated results were nondetect. No qualifiers. |
| 8149 0205 4624 (L33405-3) | PFOS | 0.659 J | Associated detects within 10 times the trip blank concentration were qualified B:<br><br>RC16A-WAT-BULK-01-072920 (L33405-17)<br>RC24-WAT-BULK-01-072920 (L33405-18) |
| 8083 3513 9226 (L33405-4) | PFOSA | 0.458 J | Associated results were nondetect. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC7-WAT-BULK-02-072820 | L33405-10 | 13C4-PFBA | 26.2 | J+ |
| WL6-WAT-BULK-01-072920 | L33405-15 | 13C4-PFBA | 43.1 | J+ |
| WL9-WAT-BULK-02-073120 | L33405-16 | 13C4-PFBA | 32.1 | J+ |
| RC16A-WAT-BULK-01-072920 | L33405-17 | 13C4-PFBA | 28.2 | J+ |
| RC24-WAT-BULK-01-072920 | L33405-18 | 13C4-PFBA | 34.4 | J+ |

**MS/MSD Recoveries (Section 6Av):** MS/MSD recovery exceedances and qualifications are listed below:

| Analyte | % Recovery | Qualifiers |
|---|---|---|
| **RC7-WAT-BULK-03-072820:** | | |
| PFOA | **162/**123 | The results for sample RC7-WAT-BULK-03-072820 were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **820/234** | |
| PFHpS | **163/**102 | The detects for samples listed below were qualified as estimated biased high (J+):<br><br>RC7-WAT-BULK-01-072820<br>RC7-WAT-BULK-02-072820<br>RC7-WAT-BULK-03-072820 |
| PFOSA | **133/**124 | |
| EtFOSAA | **132/143** | |

**Field precision (Section 6Bi):** Results associated with field imprecision were qualified as estimated (UJ and J).

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **WL9-WAT-BULK-01-073120/WL9-WAT-BULK-02-073120/WL9-WAT-BULK-03-073120:** | | | | |
| 6:2 FTS | 10 BJ | 1.35 U | 4.14 | **167** |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3-WAT-BULK-01-072820 | L33405-7 | PFDoA |
| RC7-WAT-BULK-02-072820 | L33405-10 | MeFOSAA |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73969 Report date (mm/dd/yyyy): 10/8/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/3/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Only a trip blank and equipment blank reported in this SDG. 10 sediment samples listed on COC (reported in SDG DPWG74063). |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Two samples were received at an elevated temperature of 12.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The sample ID for the trip blank was updated. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | One trip blank and one equipment blank reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for summary of detects and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS: 5.68 J ngL |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (waters) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/3/2020

## Data Validation Qualifications for DPWG73969

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| 8103 4951 0246 | L33535-1 | 12.9 |
| EQ-AUGER-081820 | L33535-2 | 12.9 |

**Trip and Equipment Blank detections (Section 3Aii):** Associated results are reported in SDGs DPWG74026 and DPWG74063. Please see the data review for SDGs DPWG74026 and DPWG74063 for qualifications.

| Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| 8103 4951 0246 (L33535-1) | 6:2 FTS | 5.73 J | This trip blank detect was determined to be laboratory contamination (see method blank qualifications below). |
| EQ-AUGER-08182 (L33535-2) | PFHxA | 0.927 J | See SDGs DPWG74026 and DPWG74063 for qualifications. |
| | PFHpA | 0.574 J | |
| | PFOA | 0.624 J | |
| | PFNA | 0.767 J | |
| | PFDA | 0.643 J | |
| | PFUnA | 0.494 J | |
| | PFOS | 0.653 J | |
| | 6:2 FTS | 87.3 J | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG73415-101 | 6:2 FTS | 5.68 J | 8103 4951 0246 (Trip Blank) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EQ-AUGER-081820 | L33535-2 | D5-N-EtFOSA | 46.3 | UJ |

**LCS and LCSD recoveries (Section 5Bi):** LCS/LCSD recovery exceedances and qualifications are listed below:

| Analyte | % Recovery | Qualifiers |
|---|---|---|
| **WG73415-102/WG73415-103:** | | |
| 6:2 FTS | **155/135** | Associated results qualified as estimated biased high (J+):<br>8103 4951 0246 (L33535-1)<br>EQ-AUGER-081820 (L33535-2) |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EQ-AUGER-081820 | L33535-2 | PFNA |
| EQ-AUGER-081820 | L33535-2 | PFUnA |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG73996 Report date (mm/dd/yyyy): 10/9/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/3/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples were received at an elevated temperature of 11.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank (L33501-1) and equipment blank (L33501-2) listed on COCs; however, not reported in this SDG (reported in DPWG74065). |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | Trip blank - no detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS: 0.767 ngL<br>PFOSA: 0.049 J ngL |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS sed/soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | 6:2 FTS 218%<br>Results were previously qualified due to method blanks. No further qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | FC2-SED-0-6-03-081420 (L33499-27 (A)) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (soil/sed) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/3/2020

## Data Validation Qualifications for DPWG73996

**Chain of custody preservation, and holding time (Section 1C):** All samples were received at a temperature of 11.8 °C. Detects were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Equipment Blank detections (Section 3Aii):** Detects within 10 times the equipment blank concentration were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| EQ-AUGER-081420 (L33501-2) | PFOS | 2.59 J | FC2A-SED-0-6-01-081420 (L33499-22)<br>FC2-SED-0-6-02-081420 (L33499-26) |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG73342-101 | 6:2 FTS | 0.767 | RC21-SED-BANK-WEST-0-6-01-081320 (L33499-15)<br>RC18B-SED-WET-0-6-01-081220 (L33499-16)<br>RC18C-SED-WET-0-6-01-081220 (L33499-17)<br>EP17A-SED-WET-0-6-01-081220 (L33499-18)<br>EP17B-SED-WET-0-6-01-081220 (L33499-19)<br>EP17C-SED-WET-0-6-01-081220 (L33499-20)<br>FC2-SED-0-6-01-081420 (L33499-21)<br>FC2A-SED-0-6-01-081420 (L33499-22)<br>VB3-SED-0-6-01-081320 (L33499-23)<br>VB3-SED-BANK-SOUTH-0-6-01-081320 (L33499-24)<br>VB3-SED-BANK-SOUTH-0-6-02-081320 (L33499-25)<br>FC2-SED-0-6-02-081420 (L33499-26)<br>FC2-SED-0-6-03-081420 (L33499-27 (A)) |
| | PFOSA | 0.049 J | RC21-SED-BANK-WEST-0-6-01-081320 (L33499-15)<br>RC18B-SED-WET-0-6-01-081220 (L33499-16)<br>EP17B-SED-WET-0-6-01-081220 (L33499-19) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC21-SED-BANK-WEST-0-6-01-081320 | L33499-15 | D3-N-MeFOSA | 43.3 | UJ |
| RC21-SED-BANK-WEST-0-6-01-081320 | L33499-15 | D5-N-EtFOSA | 34.6 | UJ |
| RC18B-SED-WET-0-6-01-081220 | L33499-16 | D3-N-MeFOSA | 45.1 | UJ |
| RC18B-SED-WET-0-6-01-081220 | L33499-16 | D5-N-EtFOSA | 35.5 | UJ |
| RC18C-SED-WET-0-6-01-081220 | L33499-17 | D3-N-MeFOSA | 43.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18C-SED-WET-0-6-01-081220 | L33499-17 | D5-N-EtFOSA | 28.4 | UJ |
| EP17A-SED-WET-0-6-01-081220 | L33499-18 | D5-N-EtFOSA | 35.2 | UJ |
| EP17B-SED-WET-0-6-01-081220 | L33499-19 | D3-N-MeFOSA | 44.5 | UJ |
| EP17B-SED-WET-0-6-01-081220 | L33499-19 | D5-N-EtFOSA | 34.7 | UJ |
| EP17C-SED-WET-0-6-01-081220 | L33499-20 | D3-N-MeFOSA | 49.6 | UJ |
| EP17C-SED-WET-0-6-01-081220 | L33499-20 | D5-N-EtFOSA | 40.3 | UJ |
| FC2-SED-0-6-01-081420 | L33499-21 | D5-N-EtFOSA | 49.4 | UJ |
| FC2A-SED-0-6-01-081420 | L33499-22 | D3-N-MeFOSA | 47.1 | UJ |
| FC2A-SED-0-6-01-081420 | L33499-22 | D5-N-EtFOSA | 33.3 | UJ |
| VB3-SED-0-6-01-081320 | L33499-23 | D3-N-MeFOSA | 42.9 | UJ |
| VB3-SED-0-6-01-081320 | L33499-23 | D5-N-EtFOSA | 33.5 | UJ |
| VB3-SED-BANK-SOUTH-0-6-01-081320 | L33499-24 | D5-N-EtFOSA | 45.8 | UJ |
| VB3-SED-BANK-SOUTH-0-6-02-081320 | L33499-25 | D3-N-MeFOSA | 36.9 | UJ |
| VB3-SED-BANK-SOUTH-0-6-02-081320 | L33499-25 | D5-N-EtFOSA | 27.4 | UJ |
| FC2-SED-0-6-02-081420 | L33499-26 | D5-N-EtFOSA | 45.6 | UJ |
| FC2-SED-0-6-03-081420 | L33499-27 (A) | D3-N-MeFOSA | 43.0 | UJ |
| FC2-SED-0-6-03-081420 | L33499-27 (A) | D5-N-EtFOSA | 27.9 | UJ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74026 Report date (mm/dd/yyyy): 10/19/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/4/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | The sample was received at an elevated temperature of 12.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank (L33535-1) listed on COCs; however, not reported in this SDG. This trip blank was reported in DPWG73969.<br>The associated equipment blank EQ-AUGER-081820 was reported in SDG DPWG73969. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Trip Blank 8103 4951 0246 (L33535-1):<br>6:2 FTS 5.73 BJ ng/L<br>This trip blank detect was previously determined to be laboratory contamination. No qualifiers. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications due to equipment blank results. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS sed/soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | 6:2 FTS 166%<br>Associated results were nondetect. No qualifications required. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☒ | ☐ | ☐ | 50% limit for PFAS (soil/sed) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/4/2020

## Data Validation Qualifications for DPWG74026

**Chain of custody preservation, and holding time (Section 1C):** The sample was received at a temperature of 12.9 °C. Detects for sample EP27C-SED-WET-0-6-01-081820 (L33534-21) were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Equipment Blank detections (Section 3Aii):** Detects within 10 times the equipment blank concentration were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| EQ-AUGER-08182 (L33535-2) | PFHxA | 0.927 J | EP27C-SED-WET-0-6-01-081820 (L33534-21) |
| | PFHpA | 0.574 J | EP27C-SED-WET-0-6-01-081820 (L33534-21) |
| | PFOA | 0.624 J | EP27C-SED-WET-0-6-01-081820 (L33534-21) |
| | PFDA | 0.643 J | EP27C-SED-WET-0-6-01-081820 (L33534-21) |
| | PFOS | 0.653 J | Associated sample concentration greater than 10 time the blank concentration. No qualifiers. |
| | PFNA | 0.767 J | The associated samples results were nondetect. No qualifiers. |
| | PFUnA | 0.494 J | |
| | 6:2 FTS | 87.3 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists:

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27C-SED-WET-0-6-01-081820 | L33534-21 | D3-N-MeFOSA | 42.7 | J+ |
| EP27C-SED-WET-0-6-01-081820 | L33534-21 | D5-N-EtFOSA | 32.7 | UJ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74063 Report date (mm/dd/yyyy): 10/19/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/5/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | The sample was received at an elevated temperature of 12.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank (L33535-1) listed on COCs; however, not reported in this SDG. This trip blank was reported in DPWG73969.<br>The associated equipment blank EQ-AUGER-081820 was reported in SDG DPWG73969. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 8103 4951 0246 ( L33535-1):<br>6:2 FTS 5.73 BJ ng/L<br>This trip blank detect was previously determined to be laboratory contamination. No qualifiers. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications due to equipment blank results. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 1.12 ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. Five results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed/soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 50% limit for PFAS (soil/sed) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Five samples were prepared using only 2 grams, and limits were above requirements.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Five results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/5/2020

## Data Validation Qualifications for DPWG74063

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 12.9 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Equipment Blank detections (Section 3Aii):** Detects within 10 times the equipment blank concentration were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| EQ-AUGER-08182 (L33535-2) | PFHxA | 0.927 J | EP27A-SED-0-6-01-081820 (L33534-1)<br>EP27B-SED-0-6-01-081820 (L33534-2)<br>EP1A-SED-0-6-01-081820 (L33534-3)<br>EP2A-SED-WET-0-6-01-081820 (L33534-4)<br>EP2C-SED-0-6-01-081820 (L33534-5)<br>EP18D-SED-0-6-01-081820 (L33534-7)<br>EP18A-SED-WET-0-6-01-081820 (L33534-8)<br>EP18C-SED-0-6-01-081820 (L33534-10) |
| | PFHpA | 0.574 J | |
| | PFOA | 0.624 J | EP27B-SED-0-6-01-081820 (L33534-2)<br>EP2A-SED-WET-0-6-01-081820 (L33534-4)<br>EP2B-SED-WET-0-6-01-081820 (L33534-6)<br>EP18A-SED-WET-0-6-01-081820 (L33534-8)<br>EP18C-SED-0-6-01-081820 (L33534-10)<br>EP18B-SED-WET-0-6-01-081820 (L33534-9) |
| | PFDA | 0.643 J | EP27A-SED-0-6-01-081820 (L33534-1)<br>EP27B-SED-0-6-01-081820 (L33534-2)<br>EP1A-SED-0-6-01-081820 (L33534-3)<br>EP2A-SED-WET-0-6-01-081820 (L33534-4)<br>EP2C-SED-0-6-01-081820 (L33534-5)<br>EP2B-SED-WET-0-6-01-081820 (L33534-6)<br>EP18D-SED-0-6-01-081820 (L33534-7)<br>EP18A-SED-WET-0-6-01-081820 (L33534-8)<br>EP18B-SED-WET-0-6-01-081820 (L33534-9)<br>EP18C-SED-0-6-01-081820 (L33534-10) |
| | PFOS | 0.653 J | Associated sample concentration greater than 10 time the blank concentration. No qualifiers. |
| | PFNA | 0.767 J | EP27A-SED-0-6-01-081820 (L33534-1)<br>EP2C-SED-0-6-01-081820 (L33534-5)<br>EP18D-SED-0-6-01-081820 (L33534-7)<br>EP18A-SED-WET-0-6-01-081820 (L33534-8)<br>EP18C-SED-0-6-01-081820 (L33534-10) |
| | PFUnA | 0.494 J | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | 87.3 J | Results were qualified due to method blanks (see below). |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentration were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG73418-101 | 6:2 FTS | 1.12 | EP27B-SED-0-6-01-081820 (L33534-2)<br>EP1A-SED-0-6-01-081820 (L33534-3)<br>EP18A-SED-WET-0-6-01-081820 (L33534-8)<br>WL21-SED-6-12-01-081920 (L33534-12)<br>WL23-SED-6-12-01-081920 (L33534-14)<br>WL22-SED-0-6-01-081920 (L33534-15)<br>EP28-SED-0-6-01-081920 (L33534-17) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Five results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27A-SED-0-6-01-081820 | L33534-1 | D3-N-MeFOSA | 24.5 | J+ |
| EP27A-SED-0-6-01-081820 | L33534-1 | D5-N-EtFOSA | 16.2 | R |
| EP27A-SED-0-6-01-081820 | L33534-1 | d7-NMe-FOSE | 44.3 | UJ |
| EP27A-SED-0-6-01-081820 | L33534-1 | d9-NEt-FOSE | 39.4 | UJ |
| EP27B-SED-0-6-01-081820 | L33534-2 | D3-N-MeFOSA | 46.7 | UJ |
| EP27B-SED-0-6-01-081820 | L33534-2 | D5-N-EtFOSA | 35.8 | UJ |
| EP1A-SED-0-6-01-081820 | L33534-3 | D3-N-MeFOSA | 26.8 | J+ |
| EP1A-SED-0-6-01-081820 | L33534-3 | D5-N-EtFOSA | 19.6 | R |
| EP1A-SED-0-6-01-081820 | L33534-3 | d7-NMe-FOSE | 45.1 | UJ |
| EP1A-SED-0-6-01-081820 | L33534-3 | d9-NEt-FOSE | 39.2 | UJ |
| EP2A-SED-WET-0-6-01-081820 | L33534-4 | D3-N-MeFOSA | 46.7 | UJ |
| EP2A-SED-WET-0-6-01-081820 | L33534-4 | D5-N-EtFOSA | 34.3 | UJ |
| EP2C-SED-0-6-01-081820 | L33534-5 | 13C2-PFTeDA | 44.0 | UJ |
| EP2C-SED-0-6-01-081820 | L33534-5 | D3-N-MeFOSA | 28.5 | J+ |
| EP2C-SED-0-6-01-081820 | L33534-5 | D5-N-EtFOSA | 19.3 | R |
| EP2C-SED-0-6-01-081820 | L33534-5 | d7-NMe-FOSE | 49.6 | UJ |
| EP2C-SED-0-6-01-081820 | L33534-5 | d9-NEt-FOSE | 42.6 | UJ |
| EP2B-SED-WET-0-6-01-081820 | L33534-6 | D5-N-EtFOSA | 45.2 | UJ |
| EP18D-SED-0-6-01-081820 | L33534-7 | D3-N-MeFOSA | 40.9 | J+ |
| EP18D-SED-0-6-01-081820 | L33534-7 | D5-N-EtFOSA | 29.5 | UJ |
| EP18D-SED-0-6-01-081820 | L33534-7 | d9-NEt-FOSE | 49.4 | UJ |
| EP18A-SED-WET-0-6-01-081820 | L33534-8 | D3-N-MeFOSA | 37.6 | UJ |
| EP18A-SED-WET-0-6-01-081820 | L33534-8 | D5-N-EtFOSA | 26.9 | UJ |
| EP18A-SED-WET-0-6-01-081820 | L33534-8 | d9-NEt-FOSE | 49.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18B-SED-WET-0-6-01-081820 | L33534-9 | D5-N-EtFOSA | 46.7 | UJ |
| EP18C-SED-0-6-01-081820 | L33534-10 | D3-N-MeFOSA | 26.8 | J+ |
| EP18C-SED-0-6-01-081820 | L33534-10 | D5-N-EtFOSA | 18.2 | R |
| EP18C-SED-0-6-01-081820 | L33534-10 | d7-NMe-FOSE | 49.5 | UJ |
| EP18C-SED-0-6-01-081820 | L33534-10 | d9-NEt-FOSE | 42.0 | UJ |
| WL21-SED-0-6-01-081920 | L33534-11 | 13C2-PFTeDA | 37.2 | UJ |
| WL21-SED-0-6-01-081920 | L33534-11 | D3-N-MeFOSA | 26.9 | UJ |
| WL21-SED-0-6-01-081920 | L33534-11 | D5-N-EtFOSA | 18.2 | R |
| WL21-SED-0-6-01-081920 | L33534-11 | d7-NMe-FOSE | 38.0 | UJ |
| WL21-SED-0-6-01-081920 | L33534-11 | d9-NEt-FOSE | 31.8 | UJ |
| EP28-SED-0-6-01-081920 | L33534-17 | D5-N-EtFOSA | 45.5 | UJ |
| EP26A-SED-WET-0-6-01-081820 | L33534-18 | D5-N-EtFOSA | 35.9 | UJ |
| EP26A-SED-WET-0-6-01-081820 | L33534-18 | d7-NMe-FOSE | 25.6 | UJ |
| EP26A-SED-WET-0-6-01-081820 | L33534-18 | d9-NEt-FOSE | 48.0 | UJ |
| EP26B-SED-0-6-01-081820 | L33534-19 | D3-N-MeFOSA | 34.4 | J+ |
| EP26B-SED-0-6-01-081820 | L33534-19 | D5-N-EtFOSA | 24.7 | UJ |
| EP26B-SED-0-6-01-081820 | L33534-19 | d9-NEt-FOSE | 47.5 | UJ |
| EP26C-SED-0-6-01-081820 | L33534-20 | D3-N-MeFOSA | 35.8 | J+ |
| EP26C-SED-0-6-01-081820 | L33534-20 | D5-N-EtFOSA | 23.0 | UJ |
| EP26C-SED-0-6-01-081820 | L33534-20 | d9-NEt-FOSE | 49.1 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP1A-SED-0-6-01-081820 | L33534-3 | N-MeFOSA |
| EP2A-SED-WET-0-6-01-081820 | L33534-4 | PFHpS |
| EP2C-SED-0-6-01-081820 | L33534-5 | N-MeFOSA |
| EP18B-SED-WET-0-6-01-081820 | L33534-9 | PFDA |
| EP18B-SED-WET-0-6-01-081820 | L33534-9 | EtFOSAA |
| WL21-SED-0-6-01-081920 | L33534-11 | PFUnA |
| WL21-SED-0-6-01-081920 | L33534-11 | EtFOSAA |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74065 Report date (mm/dd/yyyy): 10/19/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/5/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The sample was received at an elevated temperature of 11.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank 8149 0205 3710 (L33501-1) and equipment blank EQ-AUGER-081420 (L33501-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Trip blank - no detects<br>EQ-AUGER-081420 (L33501-2):<br>PFOS 2.59 ng/L<br>Samples assocaited with the equipment blank are reported in SDG DPWG73996. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | The sample concentrations were greater than 10 times the blank concentration. No qualifiers. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. One result was rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (water) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used for the foam samples.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**One result was rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/5/2020

## Data Validation Qualifications for DPWG74065

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 11.8 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP16-FOAM-01-072920 | L33501-3 | 13C9-PFNA | 36.2 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | 13C6-PFDA | 30.5 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | 13C7-PFUnA | 30.7 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | 13C2-PFDoA | 28.5 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | 13C2-PFTeDA | 19.3 | R |
| EP16-FOAM-01-072920 | L33501-3 | 13C8-PFOS | 32.2 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | 13C2-8:2 FTS | 35.1 | UJ |
| EP16-FOAM-01-072920 | L33501-3 | 13C8-PFOSA | 41.5 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | D3-N-MeFOSA | 38.1 | UJ |
| EP16-FOAM-01-072920 | L33501-3 | D5-N-EtFOSA | 32.4 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | D3-MeFOSAA | 32.5 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | D5-EtFOSAA | 44.6 | J+ |
| EP16-FOAM-01-072920 | L33501-3 | d7-NMe-FOSE | 27.2 | UJ |
| EP16-FOAM-01-072920 | L33501-3 | d9-NEt-FOSE | 22.4 | UJ |
| FP1-FOAM-01-073120 | L33501-4 | 13C2-PFDoA | 46.3 | J+ |
| FP1-FOAM-01-073120 | L33501-4 | 13C2-PFTeDA | 22.7 | UJ |
| FP1-FOAM-01-073120 | L33501-4 | d7-NMe-FOSE | 48.7 | UJ |
| FP1-FOAM-01-073120 | L33501-4 | d9-NEt-FOSE | 35.1 | UJ |

**LCS and LCSD recoveries (Section 5Bi):** LCS/LCSD recovery exceedances and qualifications are listed below:

| Analyte | % Recovery | Qualifiers |
|---|---|---|
| **WG73319-102 (A)/ WG73319-103:** | | |
| MeFOSAA | **131**/119 | Associated detects were qualified as estimated biased high (J+):<br>EP16-FOAM-01-072920 (L33501-3)<br>FP1-FOAM-01-073120 (L33501-4) |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP16-FOAM-01-072920 | L33501-3 | PFBS |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74066_3 Report date (mm/dd/yyyy): 12/15/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/26/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at an elevated temperature of 6.5 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks (L33405-1), (L33405-2), (L33405-3), and (L33405-4), equipment blanks (L33405-5), and (L33405-6) reported in DPWG73897. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | L33405-1 - nondetect<br>L33405-2:<br>6:2 FTS 1.65 J ng/L<br>L33405-3:<br>6:2 FTS 0.659 J ng/L<br>L33405-4:<br>PFOSA 0.458 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 12 ng/L<br>PFOSA 0.506 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. Eleven results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | WL9-WAT-BULK-03-073120 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFOS 61.1%/52.4%<br>The result for sample WL9-WAT-BULK-03-073120 was greater than 4 times the spike concentration. No qualifiers. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD<br>FD: WL9-WAT-BULK-01-073120/WL9-WAT-BULK-03-073120 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water)<br>Results attributable to laboratory contamination were not evaluated. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | The laboratory noted that the POS result for sample RC25-WAT-BULK-01-072920 (SGS AXYS ID: L33405-36) was reported from a re-injection due to carryover during the original analyses. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Eleven results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/26/2020

## Data Validation Qualifications for DPWG74066_3

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperature of 6.5 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID |
|---|---|
| EP23-WAT-BULK-01-072920 | L33405-20 |
| WL12-WAT-BULK-01-073120 | L33405-21 |
| WL15-WAT-BULK-01-073120 | L33405-22 |
| VB1-WAT-BULK-01-073120 | L33405-23 |
| VB3-WAT-BULK-01-073120 | L33405-24 |
| RC25-WAT-BULK-01-072920 | L33405-36 |

**Trip Blank detections (Section 3Aii):** Associated detects within 10 times the trip blank concentrations were qualified B.

| Trip Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| 8083 3513 9237 (L33405-2) | 6:2 FTS | 1.65 J | Sample results were qualified due to method blanks (see below) |
| 8149 0205 4624 (L33405-3) | PFOS | 0.659 J | VB1-WAT-BULK-01-073120 (L33405-23)<br>RC25-WAT-BULK-01-072920 (L33405-36 i) |
| 8083 3513 9226 (L33405-4) | PFOSA | 0.458 J | Sample results were qualified due to method blanks (see below) |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG73148-101 | 6:2 FTS | 12.0 | EP23-WAT-BULK-01-072920 (L33405-20)<br>WL12-WAT-BULK-01-073120 (L33405-21)<br>WL15-WAT-BULK-01-073120 (L33405-22)<br>VB1-WAT-BULK-01-073120 (L33405-23)<br>VB3-WAT-BULK-01-073120 (L33405-24)<br>EP8-WAT-BULK-01-072920 (L33405-25)<br>EP16-WAT-BULK-01-072920 (L33405-26)<br>EP9-WAT-BULK-01-072920 (L33405-27)<br>EP19-WAT-BULK-01-072920 (L33405-28)<br>EP21-WAT-BULK-01-072920 (L33405-29)<br>FP1-WAT-BULK-01-072920 (L33405-30)<br>WL9-WAT-BULK-03-073120 (L33405-32 (A))<br>RC22-WAT-BULK-01-072820 (L33405-33)<br>EP24-WAT-BULK-01-072820 (L33405-34)<br>WL9-WAT-BULK-01-073120 (L33405-35) |

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| | | | RC25-WAT-BULK-01-072920 (L33405-36)<br>EP18-WAT-BULK-01-072920 (L33405-37) |
| | PFOSA | 0.506 J | EP23-WAT-BULK-01-072920 (L33405-20)<br>WL12-WAT-BULK-01-073120 (L33405-21)<br>WL15-WAT-BULK-01-073120 (L33405-22)<br>EP8-WAT-BULK-01-072920 (L33405-25)<br>EP16-WAT-BULK-01-072920 (L33405-26)<br>EP9-WAT-BULK-01-072920 (L33405-27)<br>EP19-WAT-BULK-01-072920 (L33405-28)<br>EP21-WAT-BULK-01-072920 (L33405-29)<br>WL9-WAT-BULK-03-073120 (L33405-32 (A))<br>WL9-WAT-BULK-01-073120 (L33405-35)<br>EP18-WAT-BULK-01-072920 (L33405-37) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Eleven results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP23-WAT-BULK-01-072920 | L33405-20 | 13C4-PFBA | 5.91 | R |
| WL12-WAT-BULK-01-073120 | L33405-21 | 13C4-PFBA | 13.5 | R |
| WL15-WAT-BULK-01-073120 | L33405-22 | 13C4-PFBA | 9.66 | R |
| VB1-WAT-BULK-01-073120 | L33405-23 | 13C4-PFBA | 45.3 | J+ |
| VB3-WAT-BULK-01-073120 | L33405-24 | 13C4-PFBA | 27.4 | J+ |
| EP8-WAT-BULK-01-072920 | L33405-25 | 13C4-PFBA | 45.5 | J+ |
| EP16-WAT-BULK-01-072920 | L33405-26 | 13C4-PFBA | 15.3 | R |
| EP9-WAT-BULK-01-072920 | L33405-27 | 13C4-PFBA | 9.51 | R |
| EP19-WAT-BULK-01-072920 | L33405-28 | 13C4-PFBA | 28.0 | J+ |
| EP21-WAT-BULK-01-072920 | L33405-29 | 13C4-PFBA | 12.4 | R |
| FP1-WAT-BULK-01-072920 | L33405-30 | 13C4-PFBA | 17.8 | R |
| WL9-WAT-BULK-03-073120 | L33405-32 (A) | 13C4-PFBA | 14.7 | R |
| RC22-WAT-BULK-01-072820 | L33405-33 | 13C4-PFBA | 28.5 | J+ |
| EP24-WAT-BULK-01-072820 | L33405-34 | 13C4-PFBA | 2.67 | R |
| EP24-WAT-BULK-01-072820 | L33405-34 | 13C5-PFPeA | 31.0 | J+ |
| WL9-WAT-BULK-01-073120 | L33405-35 | 13C4-PFBA | 14.6 | R |
| EP18-WAT-BULK-01-072920 | L33405-37 | 13C4-PFBA | 4.47 | R |
| EP18-WAT-BULK-01-072920 | L33405-37 | 13C5-PFPeA | 48.6 | J+ |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74067_2 Report date (mm/dd/yyyy): 11/14/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/6/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | The bottle for one sample VB3-WAT-BULK-01-082620 (L33576-12) was received without labels. |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at an elevated temperature of 8.1 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The IDs for the trip blanks were updated. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Two trip blanks:<br>3964 5096 5149 (L33576-1)<br>3964 5118 2488 (L33576-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. Four results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD<br>FD: EP21-WAT-BULK-01-082620/EP21-WAT-BULK-03-082620 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Four results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/6/2020

## Data Validation Qualifications for DPWG74067

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperature of 8.1 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID |
|---|---|
| 3964 5096 5149 | L33576-1 |
| RC3-WAT-BULK-01-082620 | L33576-3 |
| RC7-WAT-BULK-01-082620 | L33576-4 |
| RC14-WAT-BULK-01-082620 | L33576-5 |
| RC21-WAT-BULK-01-082620 | L33576-6 |
| RC18-WAT-BULK-01-082620 | L33576-7 |
| EP19-WAT-BULK-01-082620 | L33576-8 |
| EP21-WAT-BULK-01-082620 | L33576-10 |
| EP16-WAT-BULK-01-082620 | L33576-11 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC7-WAT-BULK-01-082620 | L33576-4 | 13C4-PFBA | 37.4 | J+ |
| RC14-WAT-BULK-01-082620 | L33576-5 | 13C4-PFBA | 25.6 | J+ |
| RC21-WAT-BULK-01-082620 | L33576-6 | 13C4-PFBA | 49.3 | J+ |
| RC18-WAT-BULK-01-082620 | L33576-7 | 13C4-PFBA | 15.1 | R |
| EP19-WAT-BULK-01-082620 | L33576-8 | 13C4-PFBA | 39.9 | J+ |
| EP21-WAT-BULK-01-082620 | L33576-10 | 13C4-PFBA | 12.4 | R |
| EP16-WAT-BULK-01-082620 | L33576-11 | 13C4-PFBA | 13.5 | R |
| VB3-WAT-BULK-01-082620 | L33576-12 | 13C4-PFBA | 34.3 | J+ |
| EP21-WAT-BULK-02-082620 | L33576-13 | 13C4-PFBA | 31.6 | J+ |
| WL6-WAT-BULK-01-082620 | L33576-15 | 13C4-PFBA | 27.2 | J+ |
| WL15-WAT-BULK-01-082620 | L33576-16 | 13C4-PFBA | 8.69 | R |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP21-WAT-BULK-02-082620 | L33576-13 | PFNA |
| WL15-WAT-BULK-01-082620 | L33576-16 | EtFOSAA |

Any R qualifiers take precedence over other qualifiers.  Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74111 Report date (mm/dd/yyyy): 10/20/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/6/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at an elevated temperature of 10.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | trip blank (L33614-2) not reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | Could not review trip blank data. Not reported. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/6/2020

## Data Validation Qualifications for DPWG74111

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 10.8 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15-WAT-BULK-01-090420 | L33614-21 | 13C4-PFBA | 36.0 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74112 Report date (mm/dd/yyyy): 10/20/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/6/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at an elevated temperature of 8.9 and 6.5 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks (L33383-1), (L33405-1), (L33405-2), and (L33405-3) not reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TB L33383-1 reported in DPWG73712:<br>L33405 TBs reported in DPWG73897.<br>See data qualifications summary for trip blank detects. No qualifiers. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. One result was rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**One result was rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/6/2020

## Data Validation Qualifications for DPWG74112

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperatures greater than 6 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID | Temperature (°C) |
|---|---|---|
| MW13B-GW-295-305-01-072320 | L33384-1 R | 8.9 |
| MW9A-GW-140-150-01-072420 | L33384-2 R | 8.9 |
| MW2A-GW-240-263-01-072420 | L33384-3 R | 8.9 |
| RC25-WAT-BULK-01-072920 | L33405-36 R | 6.5 |

**Trip Blank detections (Section 3Aii):** Results did not require qualification per table below.

| Trip Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| 809964326003 TB L33383-1 | PFOSA | 1.11 J | This analyte was not reported for SDG DPWG74112. No qualifiers. |
| 8083 3513 9237 (L33405-2) | 6:2 FTS | 1.65 J | This analyte was not reported for SDG DPWG74112. No qualifiers. |
| 8149 0205 4624 (L33405-3) | PFOS | 0.659 J | Associated detects (sample RC25-WAT-BULK-01-072920) were greater than 10 times the trip blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13B-GW-295-305-01-072320 | L33384-1 R | 13C4-PFBA | 27.8 | J+ |
| MW9A-GW-140-150-01-072420 | L33384-2 R | 13C4-PFBA | 8.41 | R |
| MW9A-GW-140-150-01-072420 | L33384-2 R | 13C2-PFTeDA | 48.1 | UJ |
| MW2A-GW-240-263-01-072420 | L33384-3 R | 13C4-PFBA | 48.7 | J+ |
| RC22-WAT-BULK-01-072820 | L33405-33 R | 13C4-PFBA | 43.1 | J+ |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74129 Report date (mm/dd/yyyy): 11/5/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/27/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | Some of the samples were received at an elevated temperature of 10.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | The IDs for the trip blanks were updated. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Two trip blanks:<br>3966 5179 9945 (L33614-1)<br>3966 5179 9956 (L33614-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 3966 5179 9956 (L33614-2):<br>6:2 FTS 1.48 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. Two results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Two results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/27/2020

## Data Validation Qualifications for DPWG74129

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperature of 10.8 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID |
|---|---|
| 3966 5179 9956 | L33614-2 |
| EP16-WAT-BULK-01-090120 | L33614-13 |
| WL6-WAT-BULK-01-090120 | L33614-14 |
| WL15-WAT-BULK-01-090120 | L33614-15 |
| VB3-WAT-BULK-01-090120 | L33614-16 |
| RC3-WAT-BULK-01-090420 | L33614-17 |
| RC21-WAT-BULK-01-090420 | L33614-18 |
| EP16-WAT-BULK-01-090420 | L33614-19 |
| EP19-WAT-BULK-01-090420 | L33614-20 |

**Trip Blank detections (Section 3Aii):** Associated detects within 10 times the trip blank concentrations were qualified B.

| Trip Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| 3966 5179 9956 (L33614-2) | 6:2 FTS | 1.48 J | EP16-WAT-BULK-01-090120 (L33614-13)<br>WL15-WAT-BULK-01-090120 (L33614-15)<br>EP16-WAT-BULK-01-090420 (L33614-19) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-WAT-BULK-01-090120 | L33614-7 | 13C4-PFBA | 13.8 | R |
| RC21-WAT-BULK-01-090120 | L33614-8 | 13C4-PFBA | 27.9 | J+ |
| EP19-WAT-BULK-01-090120 | L33614-11 | 13C4-PFBA | 42.9 | J+ |
| EP21-WAT-BULK-01-090120 | L33614-12 | 13C4-PFBA | 23.1 | J+ |
| EP16-WAT-BULK-01-090120 | L33614-13 | 13C4-PFBA | 26.9 | J+ |
| WL6-WAT-BULK-01-090120 | L33614-14 | 13C4-PFBA | 46.1 | J+ |
| WL15-WAT-BULK-01-090120 | L33614-15 | 13C4-PFBA | 20.2 | J+ |
| RC21-WAT-BULK-01-090420 | L33614-18 | 13C4-PFBA | 18.1 | R |
| EP16-WAT-BULK-01-090420 | L33614-19 | 13C4-PFBA | 32.3 | J+ |
| EP19-WAT-BULK-01-090420 | L33614-20 | 13C4-PFBA | 29.1 | J+ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3-WAT-BULK-02-090120 | L33614-4 | MeFOSAA |
| RC14-WAT-BULK-01-090120 | L33614-6 | PFOS |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist


Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560


*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74278 Report date (mm/dd/yyyy): 10/30/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/6/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additiional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The sample was received at an elevated temperature of 12.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank (L33535-1) listed on COCs; however, not reported in this SDG. This trip blank was reported in DPWG73969. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | 8103 4951 0246 ( L33535-1):<br>6:2 FTS 5.73 BJ ng/L<br>This detect was previously determined to be laboratory contamination. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed/soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/6/2020

## Data Validation Qualifications for DPWG74278

**Chain of custody preservation, and holding time (Section 1C):** The sample was received at a temperature of 12.9 °C. Detects for sample WL23-SED-0-6-01-081920 (L33534-13 R) were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74445 Report date (mm/dd/yyyy): 11/12/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/6/2020

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | The sample were received at an elevated temperatures. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Receipt temperature exceedances are discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed<br>ICAL PFTrDA CS2 and CS3 standard exceedances<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks (listed on COCs, not reported in this SDG):<br>393509322683 (L33145-1) reported in EDD V72646PFAS<br>808335139134 (L33230-1) reported in EDD V72776PFAS<br>808335139178 (L33230-2) reported in EDD V72776PFAS<br>8149 0205 3710 (L33501-1) reported in SDG DPWG74065 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blanks were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFPeA 9.04 ng/g<br>PFHxA 5.74 ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed/soil limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 50% limit for PFAS (soil/sed) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/6/2020

## Data Validation Qualifications for DPWG74445

**Chain of custody preservation, and holding time (Section 1C):** Results listed below were qualified (UJ and J-) due to sample receipt temperatures.

| Field Sample ID | Lab ID | Receipt Temperature (°C) |
|---|---|---|
| RC12-SED-COMP-0-6-01-042420 | L33049-1 R4i2 | 7.7 |
| RC3A-SED-WET-0-6-01-042520 | L33049-2 R4i2 | 7.7 |
| RC12-SED-0-6-01-042520 | L33049-3 R4i2 | 7.7 |
| MW13B-SOIL-COMP-46-50-01-061520 | L33232-1 R4i2 | 9.3 |
| MW13B-SOIL-COMP-121-125-01-061520 | L33232-2 R4i2 | 9.3 |
| MW2A-SOIL-11-12-01-062220 | L33232-3 R4i2 | 10.6 |
| RC20A-SED-WET-0-6-01-081220 | L33499-4 Ri2 | 11.8 |
| RC22-SED-EAST-0-6-01-081320 | L33499-6 Ri2 | 11.8 |
| EP17A-SED-WET-0-6-01-081220 | L33499-18 Ri2 | 11.8 |

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| ICAL CS2 Standard | PFTrDA | 73.2% | 75-125% | All PFTrDA results were nondetect and qualified as estimated (UJ):<br><br>RC12-SED-COMP-0-6-01-042420 (L33049-1 R4i2)<br>RC3A-SED-WET-0-6-01-042520 (L33049-2 R4i2)<br>RC12-SED-0-6-01-042520 (L33049-3 R4i2)<br>MW9A-SOIL-5-7-01-060220 (L33148-1 R4i2)<br>MW13B-SOIL-COMP-46-50-01-061520 (L33232-1 R4i2)<br>MW13B-SOIL-COMP-121-125-01-061520 (L33232-2 R4i2)<br>MW2A-SOIL-11-12-01-062220 (L33232-3 R4i2)<br>RC20A-SED-WET-0-6-01-081220 (L33499-4 Ri2)<br>RC22-SED-EAST-0-6-01-081320 (L33499-6 Ri2)<br>EP17A-SED-WET-0-6-01-081220 (L33499-18 Ri2) |
| ICAL CS3 Standard | PFTrDA | 126% | 75-125% | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74004-101 i2 | PFPeA | 9.04 | MW13B-SOIL-COMP-121-125-01-061520 (L33232-2 R4i2)<br>MW2A-SOIL-11-12-01-062220 (L33232-3 R4i2)<br>RC20A-SED-WET-0-6-01-081220 (L33499-4 Ri2)<br>RC22-SED-EAST-0-6-01-081320 (L33499-6 Ri2)<br>EP17A-SED-WET-0-6-01-081220 (L33499-18 Ri2) |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| | PFHxA | 5.74 | RC3A-SED-WET-0-6-01-042520 (L33049-2 R4i2)<br>MW13B-SOIL-COMP-121-125-01-061520 (L33232-2 R4i2)<br>MW2A-SOIL-11-12-01-062220 (L33232-3 R4i2)<br>RC20A-SED-WET-0-6-01-081220 (L33499-4 Ri2)<br>RC22-SED-EAST-0-6-01-081320 (L33499-6 Ri2)<br>EP17A-SED-WET-0-6-01-081220 (L33499-18 Ri2) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74675 Report date (mm/dd/yyyy): 12/22/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/11/2021

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Twenty-eight results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Twenty-eight results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/10/2021

## Data Validation Qualifications for DPWG74675

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 18-Nov-2020 2:02 | 3:3 FTCA | 0 | 70-130 | Only PFOS dilutions reported from 11/18/20 analyses. No qualifiers. |
| 18-Nov-2020 6:10 | NFDHA | 172 | 70-130 | |
| 18-Nov-2020 12:53 | 3:3 FTCA | 0 | 70-130 | |
| | NFDHA | 148 | 70-130 | |
| 18-Nov-2020 19:24 | 3:3 FTCA | 0.3 | 70-130 | |
| | NFDHA | 141 | 70-130 | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG74149-101 | PFUnA | 0.106 J | EP21-PRED-FIL-FOUR-01-091820 (L33776-3)<br>BP1-PRED-FIL-ONE-01-092020 (L33776-4)<br>BP1-PRED-FIL-TWO-01-092020 (L33776-5)<br>BP1-PRED-FIL-THREE-01-092020 (L33776-6)<br>BP1-PRED-FIL-FOUR-01-092020 (L33776-7)<br>BP1-PRED-FIL-FIVE-01-092020 (L33776-8)<br>BP1-PRED-FIL-SIX-01-092020 (L33776-9)<br>BP1-PRED-FIL-SEVEN-01-092020 (L33776-10)<br>BP1-PRED-FIL-EIGHT-01-091920 (L33776-11)<br>EP14-PRED-FIL-THREE-01-091820 (L33776-14)<br>EP14-PRED-FIL-TWO-01-091820 (L33776-17)<br>EP21-PRED-FIL-ONE-01-091720 (L33776-18) |
| | PFTrDA | 0.187 J | EP21-PRED-FIL-THREE-01-091820 (L33776-1)<br>EP21-PRED-PART-THREE-01-091820 (L33776-2)<br>EP21-PRED-FIL-FOUR-01-091820 (L33776-3)<br>BP1-PRED-FIL-TWO-01-092020 (L33776-5)<br>BP1-PRED-FIL-THREE-01-092020 (L33776-6)<br>BP1-PRED-FIL-FOUR-01-092020 (L33776-7)<br>BP1-PRED-FIL-FIVE-01-092020 (L33776-8)<br>BP1-PRED-FIL-SIX-01-092020 (L33776-9)<br>BP1-PRED-FIL-SEVEN-01-092020 (L33776-10)<br>BP1-PRED-FIL-EIGHT-01-091920 (L33776-11)<br>BP1-PRED-PART-ONE-01-092020 (L33776-12)<br>BP1-PRED-PART-SIX-01-092020 (L33776-13)<br>EP14-PRED-FIL-THREE-01-091820 (L33776-14)<br>EP14-PRED-PART-THREE-01-091820 (L33776-15)<br>EP14-PRED-PART-TWO-01-091820 (L33776-16)<br>EP14-PRED-FIL-TWO-01-091820 (L33776-17)<br>EP21-PRED-FIL-ONE-01-091720 (L33776-18)<br>EP21-PRED-FIL-TWO-01-091720 (L33776-19)<br>EP27C-PRED-PART-ONE-01-091520 (L33776-20) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Twenty-eight results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP21-PRED-FIL-THREE-01-091820 | L33776-1 | D5-N-EtFOSA | 38.6 | UJ |
| EP21-PRED-FIL-THREE-01-091820 | L33776-1 | d7-NMe-FOSE | 19.9 | R |
| EP21-PRED-FIL-THREE-01-091820 | L33776-1 | d9-NEt-FOSE | 27.2 | UJ |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | D3-N-MeFOSA | 37.6 | UJ |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | D5-N-EtFOSA | 23.1 | UJ |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | D5-EtFOSAA | 212 | No Qualifier |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | d7-NMe-FOSE | 2.87 | R |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | d9-NEt-FOSE | 12.7 | R |
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | 13C3-HFPO-DA | 47.6 | UJ |
| EP21-PRED-FIL-FOUR-01-091820 | L33776-3 | D5-N-EtFOSA | 43.5 | UJ |
| EP21-PRED-FIL-FOUR-01-091820 | L33776-3 | d7-NMe-FOSE | 10.6 | R |
| EP21-PRED-FIL-FOUR-01-091820 | L33776-3 | d9-NEt-FOSE | 19.9 | R |
| BP1-PRED-FIL-ONE-01-092020 | L33776-4 | d7-NMe-FOSE | 6.19 | R |
| BP1-PRED-FIL-ONE-01-092020 | L33776-4 | d9-NEt-FOSE | 25.5 | UJ |
| BP1-PRED-FIL-TWO-01-092020 | L33776-5 | 13C2-PFTeDA | 28.3 | UJ |
| BP1-PRED-FIL-TWO-01-092020 | L33776-5 | D3-N-MeFOSA | 37.7 | UJ |
| BP1-PRED-FIL-TWO-01-092020 | L33776-5 | D5-N-EtFOSA | 41.1 | UJ |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | 13C2-PFTeDA | 15.5 | R |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | D3-N-MeFOSA | 44.4 | UJ |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | D5-N-EtFOSA | 48.2 | UJ |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | d7-NMe-FOSE | 49.9 | UJ |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | d9-NEt-FOSE | 45.6 | UJ |
| BP1-PRED-FIL-FOUR-01-092020 | L33776-7 | D3-N-MeFOSA | 44.6 | UJ |
| BP1-PRED-FIL-FOUR-01-092020 | L33776-7 | d7-NMe-FOSE | 25.7 | UJ |
| BP1-PRED-FIL-FOUR-01-092020 | L33776-7 | d9-NEt-FOSE | 27.6 | UJ |
| BP1-PRED-FIL-FIVE-01-092020 | L33776-8 | d7-NMe-FOSE | 9.62 | R |
| BP1-PRED-FIL-FIVE-01-092020 | L33776-8 | d9-NEt-FOSE | 21.7 | J+ |
| BP1-PRED-FIL-SIX-01-092020 | L33776-9 | d7-NMe-FOSE | 12.5 | R |
| BP1-PRED-FIL-SIX-01-092020 | L33776-9 | d9-NEt-FOSE | 21.5 | UJ |
| BP1-PRED-FIL-SEVEN-01-092020 | L33776-10 | 13C2-PFTeDA | 14.4 | R |
| BP1-PRED-FIL-SEVEN-01-092020 | L33776-10 | D3-N-MeFOSA | 45.1 | UJ |
| BP1-PRED-FIL-SEVEN-01-092020 | L33776-10 | D5-N-EtFOSA | 42.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BP1-PRED-FIL-SEVEN-01-092020 | L33776-10 | d9-NEt-FOSE | 42.3 | UJ |
| BP1-PRED-FIL-EIGHT-01-091920 | L33776-11 | d7-NMe-FOSE | 13.8 | R |
| BP1-PRED-FIL-EIGHT-01-091920 | L33776-11 | d9-NEt-FOSE | 18.1 | R |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | 13C2-4:2 FTS | 224 | No Qualifier |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | D3-N-MeFOSA | 29.2 | UJ |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | D5-N-EtFOSA | 19.4 | R |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | d9-NEt-FOSE | 8.40 | R |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | d7-NMe-FOSE | 5.60 | R |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | 13C3-HFPO-DA | 26.3 | UJ |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | D3-N-MeFOSA | 46.6 | UJ |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | D5-N-EtFOSA | 36.4 | UJ |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | d9-NEt-FOSE | 3.44 | R |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | d7-NMe-FOSE | 13.0 | R |
| EP14-PRED-FIL-THREE-01-091820 | L33776-14 | D3-N-MeFOSA | 49.3 | UJ |
| EP14-PRED-FIL-THREE-01-091820 | L33776-14 | d9-NEt-FOSE | 33.7 | J+ |
| EP14-PRED-FIL-THREE-01-091820 | L33776-14 | d7-NMe-FOSE | 21.0 | UJ |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | 13C2-PFTeDA | 30.4 | J+ |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | D3-N-MeFOSA | 28.7 | UJ |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | D5-N-EtFOSA | 17.1 | R |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | d9-NEt-FOSE | 4.37 | R |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | d7-NMe-FOSE | 7.54 | R |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | 13C2-4:2 FTS | 204 | No Qualifier |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | D5-N-EtFOSA | 42.8 | UJ |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | D5-EtFOSAA | 210 | UJ |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | d9-NEt-FOSE | 5.93 | R |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | d7-NMe-FOSE | 8.23 | R |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | 13C3-HFPO-DA | 20.6 | UJ |
| EP14-PRED-FIL-TWO-01-091820 | L33776-17 | D3-N-MeFOSA | 46.3 | UJ |
| EP14-PRED-FIL-TWO-01-091820 | L33776-17 | D5-N-EtFOSA | 27.2 | UJ |
| EP14-PRED-FIL-TWO-01-091820 | L33776-17 | d9-NEt-FOSE | 5.04 | R |
| EP14-PRED-FIL-TWO-01-091820 | L33776-17 | d7-NMe-FOSE | 17.9 | R |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | 13C2-PFTeDA | 46.8 | J+ |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | D3-N-MeFOSA | 32.1 | UJ |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | D5-N-EtFOSA | 32.6 | UJ |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | d9-NEt-FOSE | 22.0 | J+ |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | d7-NMe-FOSE | 13.4 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP21-PRED-FIL-TWO-01-091720 | L33776-19 | d9-NEt-FOSE | 9.0 | R |
| EP21-PRED-FIL-TWO-01-091720 | L33776-19 | d7-NMe-FOSE | 23.4 | UJ |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | 13C2-PFTeDA | 49.4 | J+ |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | D3-N-MeFOSA | 36.2 | UJ |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | D5-N-EtFOSA | 26.1 | UJ |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | D5-EtFOSAA | 250 | J- |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | d9-NEt-FOSE | 3.96 | R |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | d7-NMe-FOSE | 15.3 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Results associated with high recoveries were nondetect and were acceptable without qualification.

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74149-102 (A)/ WG74149-103 (DUP WG74149-102) | N-MeFOSE | **170/178** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 5:3 FTCA | 148/**154** | 50-150 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP21-PRED-PART-THREE-01-091820 | L33776-2 | N-EtFOSE |
| EP21-PRED-FIL-FOUR-01-091820 | L33776-3 | N-EtFOSE |
| BP1-PRED-FIL-THREE-01-092020 | L33776-6 | PFDoA |
| BP1-PRED-FIL-FOUR-01-092020 | L33776-7 | PFTrDA |
| BP1-PRED-FIL-FIVE-01-092020 | L33776-8 | N-EtFOSE |
| BP1-PRED-FIL-SIX-01-092020 | L33776-9 | PFTeDA |
| BP1-PRED-FIL-SEVEN-01-092020 | L33776-10 | PFOA |
| BP1-PRED-FIL-EIGHT-01-091920 | L33776-11 | N-EtFOSE |
| BP1-PRED-PART-ONE-01-092020 | L33776-12 | N-EtFOSE |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | PFTeDA |
| BP1-PRED-PART-SIX-01-092020 | L33776-13 | N-EtFOSE |
| EP14-PRED-FIL-THREE-01-091820 | L33776-14 | N-EtFOSE |
| EP14-PRED-PART-THREE-01-091820 | L33776-15 | N-EtFOSE |
| EP14-PRED-PART-TWO-01-091820 | L33776-16 | N-EtFOSE |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP14-PRED-FIL-TWO-01-091820 | L33776-17 | N-EtFOSE |
| EP21-PRED-FIL-ONE-01-091720 | L33776-18 | N-EtFOSE |
| EP27C-PRED-PART-ONE-01-091520 | L33776-20 | N-EtFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74676 Report date (mm/dd/yyyy): 12/4/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/10/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☐ | ☒ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receiving log indicates one sample was not received. Also, the bottle labels did not match the COC for one sample. No qualifiers. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Thirty-three results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS tissue/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | 5:3 FTCA 168%/164%<br>Associated results were nondetect and were acceptable without qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% limit for PFAS (other/tissue) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Thirty-three results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/10/2020

## Data Validation Qualifications for DPWG74676

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/14/20 7:19 | NFDHA | 131 | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV 11/14/20 13:11 | 3:3 FTCA | 0% | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| | PFHpS | 0% | 70-130% | |
| | PFDA | Not Reported | 70-130% | |
| | PFDoA | Not Reported | 70-130% | |
| | PFMPA | Not Reported | 70-130% | |
| CCV 11/14/20 17:18 | PFDA | Not Reported | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| | PFDoA | Not Reported | 70-130% | |
| | PFMPA | 0% | 70-130% | |
| | 3:3 FTCA | 0.2% | 70-130% | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74150-101 i | PFUnA | 0.132 J | EP27C-PRED-FIL-ONE-01-091520( L33776-21 i)<br>EP27C-PRED-FIL-TWO-01-091520( L33776-23 i)<br>EP18-PRED-FIL-ONE-01-091520( L33776-26 i)<br>EP18-PRED-FIL-TWO-01-091520( L33776-27 i)<br>WL7-PRED-WHO-01-091720( L33776-31 i)<br>WL6-PRED-WHO-ONE-01-091720( L33776-32 i)<br>EP9A-PRED-FIL-TWO-01-091820( L33776-38 i)<br>EP9A-PRED-FIL-THREE-01-091820( L33776-39 i)<br>EP9A-PRED-FIL-FOUR-01-091820( L33776-40 i) |
| | PFTrDA | 0.149 J | EP27C-PRED-FIL-ONE-01-091520( L33776-21 i)<br>EP27C-PRED-PART-TWO-01-091520( L33776-24 i)<br>EP18-PRED-PART-ONE-01-091520( L33776-25 i)<br>EP18-PRED-FIL-ONE-01-091520( L33776-26 i)<br>EP18-PRED-PART-TWO-01-091520( L33776-28 i)<br>EP18C-PRED-PART-01-091620( L33776-29 i)<br>EP18C-PRED-FIL-01-091620( L33776-30 i)<br>WL7-PRED-WHO-01-091720( L33776-31 i)<br>WL6-PRED-WHO-ONE-01-091720( L33776-32 i)<br>WL6-PRED-WHO-TWO-01-091720( L33776-33 i)<br>WL3-PRED-WHO-01-091720( L33776-34 i)<br>WL5-PRED-WHO-01-091720( L33776-35 i)<br>EP9A-PRED-FIL-ONE-01-091820( L33776-36 i) |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| | | | EP9A-PRED-PART-ONE-01-091820( L33776-37 i)<br>EP9A-PRED-FIL-TWO-01-091820( L33776-38 i)<br>EP9A-PRED-FIL-THREE-01-091820( L33776-39 i)<br>EP9A-PRED-FIL-FOUR-01-091820( L33776-40 i) |
| | PFOS | 0.204 J | Sample concentrations were greater than 10 times the blank concentration. No qualifiers. |
| | 6:2 FTS | 0.717 J | EP27C-PRED-FIL-ONE-01-091520( L33776-21 i)<br>EP27C-PRED-FIL-TWO-01-091520( L33776-23 i)<br>EP27C-PRED-PART-TWO-01-091520( L33776-24 i)<br>EP18-PRED-FIL-ONE-01-091520( L33776-26 i)<br>EP18-PRED-FIL-TWO-01-091520( L33776-27 i)<br>EP18-PRED-PART-TWO-01-091520( L33776-28 i)<br>EP18C-PRED-PART-01-091620( L33776-29 i)<br>WL7-PRED-WHO-01-091720( L33776-31 i)<br>WL6-PRED-WHO-ONE-01-091720( L33776-32 i)<br>WL6-PRED-WHO-TWO-01-091720( L33776-33 i)<br>WL3-PRED-WHO-01-091720( L33776-34 i)<br>WL5-PRED-WHO-01-091720( L33776-35 i)<br>EP9A-PRED-FIL-ONE-01-091820( L33776-36 i)<br>EP9A-PRED-PART-ONE-01-091820( L33776-37 i)<br>EP9A-PRED-FIL-TWO-01-091820( L33776-38 i)<br>EP9A-PRED-FIL-THREE-01-091820( L33776-39 i)<br>EP9A-PRED-FIL-FOUR-01-091820( L33776-40 i) |
| | PFOSA | 0.115 J | EP27C-PRED-FIL-ONE-01-091520( L33776-21 i)<br>EP27C-PRED-FIL-TWO-01-091520( L33776-23 i)<br>EP18-PRED-FIL-ONE-01-091520( L33776-26 i)<br>EP18-PRED-FIL-TWO-01-091520( L33776-27 i)<br>EP18C-PRED-FIL-01-091620( L33776-30 i)<br>WL7-PRED-WHO-01-091720( L33776-31 i)<br>WL6-PRED-WHO-ONE-01-091720( L33776-32 i)<br>WL6-PRED-WHO-TWO-01-091720( L33776-33 i)<br>WL3-PRED-WHO-01-091720( L33776-34 i)<br>WL5-PRED-WHO-01-091720( L33776-35 i)<br>EP9A-PRED-PART-ONE-01-091820( L33776-37 i)<br>EP9A-PRED-FIL-TWO-01-091820( L33776-38 i)<br>EP9A-PRED-FIL-THREE-01-091820( L33776-39 i)<br>EP9A-PRED-FIL-FOUR-01-091820( L33776-40 i) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Thirty-three results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | 13C2-PFTeDA | 20.5 | UJ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | D3-N-MeFOSA | 32.0 | UJ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | D5-N-EtFOSA | 22.4 | UJ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | d7-NMe-FOSE | 7.50 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | d9-NEt-FOSE | 7.76 | R |
| EP27C-PRED-FIL-TWO-01-091520 | L33776-23 i | D5-N-EtFOSA | 31.6 | UJ |
| EP27C-PRED-FIL-TWO-01-091520 | L33776-23 i | d7-NMe-FOSE | 7.81 | R |
| EP27C-PRED-FIL-TWO-01-091520 | L33776-23 i | d9-NEt-FOSE | 16.7 | R |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | 13C2-PFTeDA | 39.4 | J+ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | 13C2-4:2 FTS | 224 | No Qualifier |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | D3-N-MeFOSA | 37.8 | UJ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | D5-N-EtFOSA | 33.5 | UJ |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | D5-EtFOSAA | 305 | No Qualifier |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | d7-NMe-FOSE | 6.42 | R |
| EP27C-PRED-FIL-ONE-01-091520 | L33776-24 i | d9-NEt-FOSE | 18.5 | R |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | 13C2-4:2 FTS | 220 | No Qualifier |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | 13C8-PFOSA | 225 | J- |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | D3-N-MeFOSA | 18.9 | R |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | D5-N-EtFOSA | 35.6 | UJ |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | D5-EtFOSAA | 344 | J- |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | d7-NMe-FOSE | 23.0 | UJ |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | d9-NEt-FOSE | 8.88 | R |
| EP18-PRED-FIL-ONE-01-091520 | L33776-26 i | D5-N-EtFOSA | 39.1 | UJ |
| EP18-PRED-FIL-ONE-01-091520 | L33776-26 i | D5-EtFOSAA | 209 | J- |
| EP18-PRED-FIL-ONE-01-091520 | L33776-26 i | d7-NMe-FOSE | 4.36 | R |
| EP18-PRED-FIL-ONE-01-091520 | L33776-26 i | d9-NEt-FOSE | 33.3 | UJ |
| EP18-PRED-FIL-TWO-01-091520 | L33776-27 i | d7-NMe-FOSE | 8.51 | R |
| EP18-PRED-FIL-TWO-01-091520 | L33776-27 i | d9-NEt-FOSE | 41.1 | UJ |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | 13C2-4:2 FTS | 223 | No Qualifier |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | D3-N-MeFOSA | 30.3 | UJ |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | D5-N-EtFOSA | 24.8 | UJ |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | D5-EtFOSAA | 315 | J- |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | d7-NMe-FOSE | 11.7 | R |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | d9-NEt-FOSE | 7.38 | R |
| EP18C-PRED-PART-01-091620 | L33776-29 i | 13C2-4:2 FTS | 235 | No Qualifier |
| EP18C-PRED-PART-01-091620 | L33776-29 i | D5-N-EtFOSA | 47.1 | UJ |
| EP18C-PRED-PART-01-091620 | L33776-29 i | D5-EtFOSAA | 381 | J- |
| EP18C-PRED-PART-01-091620 | L33776-29 i | d7-NMe-FOSE | 3.96 | R |
| EP18C-PRED-PART-01-091620 | L33776-29 i | d9-NEt-FOSE | 32.4 | UJ |
| EP18C-PRED-PART-01-091620 | L33776-29 i | 13C3-HFPO-DA | 49.5 | UJ |
| EP18C-PRED-FIL-01-091620 | L33776-30 i | D5-N-EtFOSA | 35.5 | UJ |
| EP18C-PRED-FIL-01-091620 | L33776-30 i | D5-EtFOSAA | 207 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18C-PRED-FIL-01-091620 | L33776-30 i | d7-NMe-FOSE | 4.51 | R |
| EP18C-PRED-FIL-01-091620 | L33776-30 i | d9-NEt-FOSE | 26.8 | UJ |
| WL7-PRED-WHO-01-091720 | L33776-31 i | 13C2-PFTeDA | 41.7 | J+ |
| WL7-PRED-WHO-01-091720 | L33776-31 i | 13C2-4:2 FTS | 223 | No Qualifier |
| WL7-PRED-WHO-01-091720 | L33776-31 i | D3-N-MeFOSA | 38.5 | UJ |
| WL7-PRED-WHO-01-091720 | L33776-31 i | D5-N-EtFOSA | 22.3 | UJ |
| WL7-PRED-WHO-01-091720 | L33776-31 i | d7-NMe-FOSE | 5.72 | R |
| WL7-PRED-WHO-01-091720 | L33776-31 i | d9-NEt-FOSE | 9.49 | R |
| WL7-PRED-WHO-01-091720 | L33776-31 i | 13C3-HFPO-DA | 23.7 | UJ |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | 13C2-PFTeDA | 30.4 | UJ |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | D3-N-MeFOSA | 29.1 | UJ |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | D5-N-EtFOSA | 17.7 | R |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | d7-NMe-FOSE | 12.1 | R |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | d9-NEt-FOSE | 6.9 | R |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | 13C2-PFTeDA | 19.1 | R |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | D3-N-MeFOSA | 36.9 | UJ |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | D5-N-EtFOSA | 22.5 | UJ |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | D5-EtFOSAA | 265 | J- |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | d7-NMe-FOSE | 9.30 | R |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | d9-NEt-FOSE | 7.73 | R |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | 13C3-HFPO-DA | 44.9 | UJ |
| WL3-PRED-WHO-01-091720 | L33776-34 i | 13C2-PFTeDA | 29.2 | J+ |
| WL3-PRED-WHO-01-091720 | L33776-34 i | 13C2-4:2 FTS | 236 | No Qualifier |
| WL3-PRED-WHO-01-091720 | L33776-34 i | D3-N-MeFOSA | 29.0 | UJ |
| WL3-PRED-WHO-01-091720 | L33776-34 i | D5-N-EtFOSA | 16.0 | R |
| WL3-PRED-WHO-01-091720 | L33776-34 i | D5-EtFOSAA | 241 | J- |
| WL3-PRED-WHO-01-091720 | L33776-34 i | d7-NMe-FOSE | 1.85 | R |
| WL3-PRED-WHO-01-091720 | L33776-34 i | d9-NEt-FOSE | 5.93 | R |
| WL5-PRED-WHO-01-091720 | L33776-35 i | 13C2-4:2 FTS | 207 | No Qualifier |
| WL5-PRED-WHO-01-091720 | L33776-35 i | 13C8-PFOSA | 205 | J- |
| WL5-PRED-WHO-01-091720 | L33776-35 i | D5-N-EtFOSA | 33.2 | UJ |
| WL5-PRED-WHO-01-091720 | L33776-35 i | D5-EtFOSAA | 281 | J- |
| WL5-PRED-WHO-01-091720 | L33776-35 i | d7-NMe-FOSE | 11.0 | R |
| WL5-PRED-WHO-01-091720 | L33776-35 i | d9-NEt-FOSE | 5.17 | R |
| EP9A-PRED-FIL-ONE-01-091820 | L33776-36 i | 13C2-PFTeDA | 41.5 | J+ |
| EP9A-PRED-FIL-ONE-01-091820 | L33776-36 i | D3-N-MeFOSA | 42.0 | UJ |
| EP9A-PRED-FIL-ONE-01-091820 | L33776-36 i | D5-N-EtFOSA | 31.0 | UJ |
| EP9A-PRED-FIL-ONE-01-091820 | L33776-36 i | d7-NMe-FOSE | 49.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9A-PRED-FIL-ONE-01-091820 | L33776-36 i | d9-NEt-FOSE | 30.1 | UJ |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | 13C2-PFTeDA | 32.6 | J+ |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | 13C2-4:2 FTS | 231 | No Qualifier |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | 13C2-6:2 FTS | 200 | J- |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | D3-N-MeFOSA | 31.8 | UJ |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | D5-N-EtFOSA | 20.4 | UJ |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | D5-EtFOSAA | 218 | J- |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | d7-NMe-FOSE | 8.36 | R |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | d9-NEt-FOSE | 7.86 | R |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | 13C3-HFPO-DA | 42.5 | UJ |
| EP9A-PRED-FIL-TWO-01-091820 | L33776-38 i | 13C2-PFTeDA | 12.1 | R |
| EP9A-PRED-FIL-THREE-01-091820 | L33776-39 i | D5-N-EtFOSA | 47.1 | UJ |
| EP9A-PRED-FIL-THREE-01-091820 | L33776-39 i | d7-NMe-FOSE | 9.28 | R |
| EP9A-PRED-FIL-THREE-01-091820 | L33776-39 i | d9-NEt-FOSE | 25.1 | UJ |
| EP9A-PRED-FIL-FOUR-01-091820 | L33776-40 i | D5-N-EtFOSA | 42.7 | UJ |
| EP9A-PRED-FIL-FOUR-01-091820 | L33776-40 i | d7-NMe-FOSE | 9.31 | R |
| EP9A-PRED-FIL-FOUR-01-091820 | L33776-40 i | d9-NEt-FOSE | 17.0 | R |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP27C-PRED-FIL-ONE-01-091520 | L33776-21 i | N-EtFOSE |
| EP27C-PRED-PART-TWO-01-091520 | L33776-24 i | PFTeDA |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | PFHpA |
| EP18-PRED-PART-ONE-01-091520 | L33776-25 i | N-EtFOSE |
| EP18-PRED-FIL-ONE-01-091520 | L33776-26 i | PFTrDA |
| EP18-PRED-PART-TWO-01-091520 | L33776-28 i | N-EtFOSE |
| WL7-PRED-WHO-01-091720 | L33776-31 i | PFTeDA |
| WL7-PRED-WHO-01-091720 | L33776-31 i | N-EtFOSE |
| WL6-PRED-WHO-ONE-01-091720 | L33776-32 i | N-EtFOSE |
| WL6-PRED-WHO-TWO-01-091720 | L33776-33 i | N-EtFOSE |
| WL3-PRED-WHO-01-091720 | L33776-34 i | N-EtFOSE |
| WL5-PRED-WHO-01-091720 | L33776-35 i | N-EtFOSE |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | EtFOSAA |
| EP9A-PRED-PART-ONE-01-091820 | L33776-37 i | N-EtFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74677 Report date (mm/dd/yyyy): 12/2/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/21/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Forty results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS tissue/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | N-MeFOSE 203%/155%<br>11Cl-PF3OUdS --/131%<br>5:3 FTCA --/164%<br>Associated results were nondetect and were acceptable without qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | 50% limit for PFAS (other/tissue) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Forty results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/21/2020

## Data Validation Qualifications for DPWG74677

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/23/20 15:58 | 3:3 FTCA | 58.4 | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| | PFDoA | Not Reported | 70-130% | |
| | PFMPA | Not Reported | 70-130% | |
| CCV 11/23/20 15:58 | 3:3 FTCA | 0.1 | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| | PFDoA | Not Reported | 70-130% | |
| | PFMPA | Not Reported | 70-130% | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74151-101 | PFUnA | 0.126 J | EP14-PRED-FIL-ONE-01-091820 (L33776-41)<br>WL14-PRED-WHO-TWO-01-091620 (L33776-43)<br>WL14-PRED-WHO-THREE-01-091620 (L33776-44)<br>WL14-PRED-WHO-FOUR-01-091620 (L33776-45)<br>EP21-FORE-FIL-ONE-01-091820 (L33784-1)<br>EP21-FORE-FIL-THREE-01-091820 (L33784-3)<br>EP21-FORE-PART-THREE-01-091820 (L33784-4)<br>EP21-FORE-FIL-SEVEN-01-091820 (L33784-8)<br>EP21-FORE-FIL-EIGHT-01-091820 (L33784-9)<br>EP21-FORE-PART-SEVEN-01-091820 (L33784-10)<br>EP21-FORE-FIL-NINE-01-091820 (L33784-11) |
| | PFTrDA | 0.153 J | EP14-PRED-FIL-ONE-01-091820 (L33776-41)<br>EP14-PRED-PART-ONE-01-091820 (L33776-42)<br>WL14-PRED-WHO-TWO-01-091620 (L33776-43)<br>WL14-PRED-WHO-THREE-01-091620 (L33776-44)<br>WL14-PRED-WHO-FOUR-01-091620 (L33776-45)<br>WL15A-PRED-WHO-ONE-01-091620 (L33776-46)<br>WL15A-PRED-WHO-FOUR-01-091620 (L33776-47)<br>WL15A-PRED-WHO-TWO-01-091620 (L33776-48)<br>WL15A-PRED-WHO-THREE-01-091620 (L33776-49)<br>EP21-FORE-FIL-ONE-01-091820 (L33784-1)<br>EP21-FORE-FIL-THREE-01-091820 (L33784-3)<br>EP21-FORE-PART-THREE-01-091820 (L33784-4)<br>EP21-FORE-FIL-SEVEN-01-091820 (L33784-8)<br>EP21-FORE-FIL-EIGHT-01-091820 (L33784-9)<br>EP21-FORE-PART-SEVEN-01-091820 (L33784-10)<br>EP21-FORE-FIL-NINE-01-091820 (L33784-11) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low

(J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Forty results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP14-PRED-FIL-ONE-01-091820 | L33776-41 | 13C2-PFTeDA | 45.6 | J+ |
| EP14-PRED-FIL-ONE-01-091820 | L33776-41 | D3-N-MeFOSA | 46.9 | UJ |
| EP14-PRED-FIL-ONE-01-091820 | L33776-41 | D5-N-EtFOSA | 30.7 | UJ |
| EP14-PRED-FIL-ONE-01-091820 | L33776-41 | d7-NMe-FOSE | 6.54 | R |
| EP14-PRED-FIL-ONE-01-091820 | L33776-41 | d9-NEt-FOSE | 17.8 | R |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | 13C2-PFTeDA | 43.3 | J+ |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | D5-N-EtFOSA | 31.2 | UJ |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | d7-NMe-FOSE | 7.68 | R |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | d9-NEt-FOSE | 11.0 | R |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | 13C3-HFPO-DA | 36.3 | UJ |
| WL14-PRED-WHO-TWO-01-091620 | L33776-43 | d7-NMe-FOSE | 8.03 | R |
| WL14-PRED-WHO-TWO-01-091620 | L33776-43 | d9-NEt-FOSE | 29.0 | UJ |
| WL14-PRED-WHO-THREE-01-091620 | L33776-44 | D5-N-EtFOSA | 47.7 | UJ |
| WL14-PRED-WHO-THREE-01-091620 | L33776-44 | D5-EtFOSAA | 233 | J- |
| WL14-PRED-WHO-THREE-01-091620 | L33776-44 | d7-NMe-FOSE | 5.82 | R |
| WL14-PRED-WHO-THREE-01-091620 | L33776-44 | d9-NEt-FOSE | 21.4 | J+ |
| WL14-PRED-WHO-FOUR-01-091620 | L33776-45 | D5-N-EtFOSA | 43.8 | UJ |
| WL14-PRED-WHO-FOUR-01-091620 | L33776-45 | D5-EtFOSAA | 211 | J- |
| WL14-PRED-WHO-FOUR-01-091620 | L33776-45 | d7-NMe-FOSE | 9.38 | R |
| WL14-PRED-WHO-FOUR-01-091620 | L33776-45 | d9-NEt-FOSE | 21.0 | J+ |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | 13C2-4:2 FTS | 226 | No Qualifier |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | D3-N-MeFOSA | 49.5 | UJ |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | D5-N-EtFOSA | 31.6 | UJ |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | D5-EtFOSAA | 224 | J- |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | d7-NMe-FOSE | 5.13 | R |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | d9-NEt-FOSE | 13.6 | R |
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | 13C2-PFTeDA | 44.6 | J+ |
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | D3-N-MeFOSA | 31.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | D5-N-EtFOSA | 19.5 | R |
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | d7-NMe-FOSE | 3.52 | R |
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | d9-NEt-FOSE | 7.72 | R |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | D3-N-MeFOSA | 27.7 | UJ |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | D5-N-EtFOSA | 16.6 | R |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | d7-NMe-FOSE | 2.10 | R |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | d9-NEt-FOSE | 7.44 | R |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | D3-N-MeFOSA | 29.7 | UJ |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | D5-N-EtFOSA | 16.5 | R |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | d7-NMe-FOSE | 2.12 | R |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | d9-NEt-FOSE | 6.18 | R |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | 13C3-HFPO-DA | 44.5 | UJ |
| EP21-FORE-FIL-ONE-01-091820 | L33784-1 | D5-N-EtFOSA | 34.4 | UJ |
| EP21-FORE-FIL-ONE-01-091820 | L33784-1 | d7-NMe-FOSE | 12.9 | R |
| EP21-FORE-FIL-ONE-01-091820 | L33784-1 | d9-NEt-FOSE | 14.8 | R |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | D3-N-MeFOSA | 37.0 | UJ |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | D5-N-EtFOSA | 27.2 | UJ |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | d7-NMe-FOSE | 3.70 | R |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | d9-NEt-FOSE | 10.5 | R |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | D3-N-MeFOSA | 37.0 | UJ |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | D5-N-EtFOSA | 34.3 | UJ |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | d7-NMe-FOSE | 3.89 | R |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | d9-NEt-FOSE | 18.9 | R |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | 13C2-4:2 FTS | 203 | No Qualifier |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | D3-N-MeFOSA | 37.3 | UJ |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | D5-N-EtFOSA | 35.2 | UJ |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | d7-NMe-FOSE | 4.81 | R |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | d9-NEt-FOSE | 25.4 | J+ |
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | D3-N-MeFOSA | 37.6 | UJ |
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | D5-N-EtFOSA | 35.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | d7-NMe-FOSE | 3.18 | R |
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | d9-NEt-FOSE | 8.36 | R |
| EP21-FORE-FIL-FIVE-01-091820 | L33784-6 | D3-N-MeFOSA | 44.6 | UJ |
| EP21-FORE-FIL-FIVE-01-091820 | L33784-6 | D5-N-EtFOSA | 37.0 | UJ |
| EP21-FORE-FIL-FIVE-01-091820 | L33784-6 | d7-NMe-FOSE | 2.57 | R |
| EP21-FORE-FIL-FIVE-01-091820 | L33784-6 | d9-NEt-FOSE | 14.1 | R |
| EP21-FORE-FIL-SIX-01-091820 | L33784-7 | D3-N-MeFOSA | 45.7 | UJ |
| EP21-FORE-FIL-SIX-01-091820 | L33784-7 | D5-N-EtFOSA | 41.9 | UJ |
| EP21-FORE-FIL-SIX-01-091820 | L33784-7 | d7-NMe-FOSE | 5.34 | R |
| EP21-FORE-FIL-SIX-01-091820 | L33784-7 | d9-NEt-FOSE | 12.7 | R |
| EP21-FORE-FIL-SEVEN-01-091820 | L33784-8 | D5-N-EtFOSA | 30.9 | UJ |
| EP21-FORE-FIL-SEVEN-01-091820 | L33784-8 | d7-NMe-FOSE | 5.15 | R |
| EP21-FORE-FIL-SEVEN-01-091820 | L33784-8 | d9-NEt-FOSE | 19.8 | R |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | 13C2-PFTeDA | 28.8 | J+ |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | D3-N-MeFOSA | 27.4 | UJ |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | D5-N-EtFOSA | 15.8 | R |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | d7-NMe-FOSE | 5.35 | R |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | d9-NEt-FOSE | 4.25 | R |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | 13C2-PFTeDA | 46.0 | J+ |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | D3-N-MeFOSA | 24.1 | UJ |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | D5-N-EtFOSA | 15.7 | R |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | d7-NMe-FOSE | 6.07 | R |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | d9-NEt-FOSE | 5.96 | R |
| EP21-FORE-FIL-NINE-01-091820 | L33784-11 | D5-N-EtFOSA | 35.6 | UJ |
| EP21-FORE-FIL-NINE-01-091820 | L33784-11 | d7-NMe-FOSE | 12.7 | R |
| EP21-FORE-FIL-NINE-01-091820 | L33784-11 | d9-NEt-FOSE | 20.7 | J+ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | PFHpA |
| EP14-PRED-PART-ONE-01-091820 | L33776-42 | N-EtFOSE |
| WL14-PRED-WHO-THREE-01-091620 | L33776-44 | N-EtFOSE |
| WL14-PRED-WHO-FOUR-01-091620 | L33776-45 | N-EtFOSE |
| WL15A-PRED-WHO-ONE-01-091620 | L33776-46 | N-EtFOSE |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | PFOA |
| WL15A-PRED-WHO-FOUR-01-091620 | L33776-47 | N-EtFOSE |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | EtFOSAA |
| WL15A-PRED-WHO-TWO-01-091620 | L33776-48 | N-EtFOSE |
| WL15A-PRED-WHO-THREE-01-091620 | L33776-49 | N-EtFOSE |
| EP21-FORE-FIL-ONE-01-091820 | L33784-1 | N-EtFOSE |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | PFTeDA |
| EP21-FORE-FIL-TWO-01-091820 | L33784-2 | N-EtFOSE |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | PFTrDA |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | MeFOSAA |
| EP21-FORE-FIL-THREE-01-091820 | L33784-3 | N-EtFOSE |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | PFHpA |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | MeFOSAA |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | N-EtFOSE |
| EP21-FORE-PART-THREE-01-091820 | L33784-4 | NFDHA |
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | PFOA |
| EP21-FORE-FIL-FOUR-01-091820 | L33784-5 | N-EtFOSE |
| EP21-FORE-FIL-FIVE-01-091820 | L33784-6 | N-EtFOSE |
| EP21-FORE-FIL-SIX-01-091820 | L33784-7 | N-EtFOSE |
| EP21-FORE-FIL-SEVEN-01-091820 | L33784-8 | N-EtFOSE |
| EP21-FORE-FIL-EIGHT-01-091820 | L33784-9 | N-EtFOSE |
| EP21-FORE-PART-SEVEN-01-091820 | L33784-10 | N-EtFOSE |
| EP21-FORE-FIL-NINE-01-091820 | L33784-11 | N-EtFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74689 Report date (mm/dd/yyyy): 12/16/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/18/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Forty-five results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other)<br>LCSD: RPDs within limits. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualifications.**

**Forty-five results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/18/2021

## Data Validation Qualifications for DPWG74689

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 24-Nov-2020 11:18 | NFDHA | 164 | 70-130 | Only PFOS reported from 11/24/20. No qualifiers. |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74153-101 | PFUnA | 0.161 J | BP1-FORE-FIL-ONE-01-092020 (L33784-12)<br>BP1-FORE-PART-ONE-01-092020 (L33784-13)<br>BP1-FORE-FIL-TWO-01-092020 (L33784-14)<br>BP1-FORE-PART-TWO-01-092020 (L33784-15)<br>RC21A-FORE-WHO-01-092120 (L33784-17)<br>RC18A-FORE-WHO-01-092120 (L33784-18)<br>WL7-FORE-WHO-01-091720 (L33784-19)<br>EP18-FORE-FIL-ONE-01-091520 (L33784-21)<br>EP15-FORE-WHO-THREE-01-091820 (L33784-23)<br>EP27C-FORE-FIL-ONE-01-091520 (L33784-24)<br>EP1A-FORE-FIL-01-091520 (L33784-27)<br>EP1A-FORE-WHO-01-091520 (L33784-29)<br>EP9A-FORE-FIL-ONE-01-091820 (L33784-30)<br>EP9A-FORE-PART-ONE-01-091820 (L33784-31) |
| | PFTrDA | 0.155 J | BP1-FORE-FIL-ONE-01-092020 (L33784-12)<br>BP1-FORE-PART-ONE-01-092020 (L33784-13)<br>BP1-FORE-PART-TWO-01-092020 (L33784-15)<br>BP1-FORE-WHO-THREE-01-091920 (L33784-16)<br>RC21A-FORE-WHO-01-092120 (L33784-17)<br>RC18A-FORE-WHO-01-092120 (L33784-18)<br>WL7-FORE-WHO-01-091720 (L33784-19)<br>EP18-FORE-PART-ONE-01-091520 (L33784-20)<br>EP18-FORE-FIL-ONE-01-091520 (L33784-21)<br>EP18-FORE-WHO-TWO-01-091520 (L33784-22)<br>EP15-FORE-WHO-THREE-01-091820 (L33784-23)<br>EP27C-FORE-PART-ONE-01-091520 (L33784-25)<br>EP27C-FORE-WHO-TWO-01-091520 (L33784-26)<br>EP1A-FORE-FIL-01-091520 (L33784-27)<br>EP1A-FORE-PART-01-091520 (L33784-28)<br>EP9A-FORE-FIL-ONE-01-091820 (L33784-30)<br>EP9A-FORE-PART-ONE-01-091820 (L33784-31) |
| | PFOS | 0.121 J | Sample concentrations were greater than ten times the blank concentration. No qualifiers. |
| | PFOSA | 0.115 J | BP1-FORE-WHO-THREE-01-091920 (L33784-16)<br>RC21A-FORE-WHO-01-092120 (L33784-17)<br>WL7-FORE-WHO-01-091720 (L33784-19)<br>EP15-FORE-WHO-THREE-01-091820 (L33784-23)<br>EP9A-FORE-PART-ONE-01-091820 (L33784-31) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Forty-five results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BP1-FORE-FIL-ONE-01-092020 | L33784-12 | D3-N-MeFOSA | 37.8 | UJ |
| BP1-FORE-FIL-ONE-01-092020 | L33784-12 | d7-NMe-FOSE | 22.3 | UJ |
| BP1-FORE-FIL-ONE-01-092020 | L33784-12 | d9-NEt-FOSE | 29.0 | UJ |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | 13C2-PFTeDA | 42.4 | J+ |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | D3-N-MeFOSA | 20.3 | UJ |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | D5-N-EtFOSA | 17.2 | R |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | d7-NMe-FOSE | 1.73 | R |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | d9-NEt-FOSE | 5.99 | R |
| BP1-FORE-FIL-TWO-01-092020 | L33784-14 | D3-N-MeFOSA | 36.9 | UJ |
| BP1-FORE-FIL-TWO-01-092020 | L33784-14 | D5-N-EtFOSA | 44.1 | UJ |
| BP1-FORE-FIL-TWO-01-092020 | L33784-14 | d7-NMe-FOSE | 47.7 | UJ |
| BP1-FORE-FIL-TWO-01-092020 | L33784-14 | d9-NEt-FOSE | 40.8 | UJ |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | 13C2-PFTeDA | 42.6 | J+ |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | D3-N-MeFOSA | 19.4 | R |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | D5-N-EtFOSA | 20.7 | UJ |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | d7-NMe-FOSE | 1.84 | R |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | d9-NEt-FOSE | 5.30 | R |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | 13C3-HFPO-DA | 40.1 | UJ |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | D3-N-MeFOSA | 24.4 | UJ |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | D5-N-EtFOSA | 27.8 | UJ |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | d7-NMe-FOSE | 7.58 | R |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | d9-NEt-FOSE | 5.61 | R |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | 13C3-HFPO-DA | 30.0 | UJ |
| RC21A-FORE-WHO-01-092120 | L33784-17 | 13C2-PFTeDA | 23.8 | J+ |
| RC21A-FORE-WHO-01-092120 | L33784-17 | D3-N-MeFOSA | 21.5 | UJ |
| RC21A-FORE-WHO-01-092120 | L33784-17 | D5-N-EtFOSA | 20.8 | UJ |
| RC21A-FORE-WHO-01-092120 | L33784-17 | d7-NMe-FOSE | 2.66 | R |
| RC21A-FORE-WHO-01-092120 | L33784-17 | d9-NEt-FOSE | 6.00 | R |
| RC18A-FORE-WHO-01-092120 | L33784-18 | 13C4-PFBA | 5.34 | R |
| RC18A-FORE-WHO-01-092120 | L33784-18 | 13C5-PFPeA | 35.9 | UJ |
| RC18A-FORE-WHO-01-092120 | L33784-18 | 13C2-PFTeDA | 13.7 | R |
| RC18A-FORE-WHO-01-092120 | L33784-18 | D3-N-MeFOSA | 25.2 | UJ |
| RC18A-FORE-WHO-01-092120 | L33784-18 | D5-N-EtFOSA | 12.4 | R |
| RC18A-FORE-WHO-01-092120 | L33784-18 | 13C3-HFPO-DA | 19.4 | R |
| WL7-FORE-WHO-01-091720 | L33784-19 | 13C2-PFTeDA | 25.9 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL7-FORE-WHO-01-091720 | L33784-19 | D3-N-MeFOSA | 18.2 | R |
| WL7-FORE-WHO-01-091720 | L33784-19 | D5-N-EtFOSA | 16.0 | R |
| WL7-FORE-WHO-01-091720 | L33784-19 | d7-NMe-FOSE | 11.1 | R |
| WL7-FORE-WHO-01-091720 | L33784-19 | d9-NEt-FOSE | 4.79 | R |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | 13C2-PFTeDA | 36.6 | J+ |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | D3-N-MeFOSA | 25.7 | UJ |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | D5-N-EtFOSA | 24.9 | UJ |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | D5-EtFOSAA | 316 | J- |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | d7-NMe-FOSE | 3.59 | R |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | d9-NEt-FOSE | 10.7 | R |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | 13C5-PFPeA | 273 | No Qualifier |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | 13C3-PFBS | 48.9 | UJ |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | D5-N-EtFOSA | 39.6 | UJ |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | D5-EtFOSAA | 216 | J- |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | d7-NMe-FOSE | 5.77 | R |
| EP18-FORE-FIL-ONE-01-091520 | L33784-21 | d9-NEt-FOSE | 22.8 | UJ |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | 13C2-4:2 FTS | 202 | No Qualifier |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | D3-N-MeFOSA | 46.2 | UJ |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | D5-N-EtFOSA | 34.3 | UJ |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | D5-EtFOSAA | 246 | J- |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | d7-NMe-FOSE | 6.01 | R |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | d9-NEt-FOSE | 17.9 | R |
| EP18-FORE-WHO-TWO-01-091520 | L33784-22 | 13C3-HFPO-DA | 27.9 | UJ |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | 13C2-PFTeDA | 27.7 | J+ |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | D3-N-MeFOSA | 17.0 | R |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | D5-N-EtFOSA | 11.0 | R |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | D5-EtFOSAA | 201 | No Qualifier |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | d9-NEt-FOSE | 3.26 | R |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | 13C2-PFTeDA | 42.0 | UJ |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | D3-N-MeFOSA | 29.5 | UJ |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | D5-N-EtFOSA | 32.3 | UJ |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | d7-NMe-FOSE | 23.4 | UJ |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | d9-NEt-FOSE | 18.7 | R |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | 13C2-PFTeDA | 23.0 | UJ |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | D3-N-MeFOSA | 28.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | D5-N-EtFOSA | 22.0 | UJ |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | D5-EtFOSAA | 301 | J- |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | d7-NMe-FOSE | 2.09 | R |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | d9-NEt-FOSE | 5.70 | R |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | 13C2-PFTeDA | 36.2 | J+ |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | D3-N-MeFOSA | 25.4 | UJ |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | D5-N-EtFOSA | 19.3 | R |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | D5-EtFOSAA | 219 | J- |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | d7-NMe-FOSE | 2.39 | R |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | d9-NEt-FOSE | 8.16 | R |
| EP1A-FORE-FIL-01-091520 | L33784-27 | D3-N-MeFOSA | 47.3 | UJ |
| EP1A-FORE-FIL-01-091520 | L33784-27 | D5-N-EtFOSA | 36.5 | UJ |
| EP1A-FORE-FIL-01-091520 | L33784-27 | d7-NMe-FOSE | 14.8 | R |
| EP1A-FORE-FIL-01-091520 | L33784-27 | d9-NEt-FOSE | 15.3 | R |
| EP1A-FORE-PART-01-091520 | L33784-28 | D3-N-MeFOSA | 24.5 | UJ |
| EP1A-FORE-PART-01-091520 | L33784-28 | D5-N-EtFOSA | 29.8 | UJ |
| EP1A-FORE-PART-01-091520 | L33784-28 | D5-EtFOSAA | 234 | J- |
| EP1A-FORE-PART-01-091520 | L33784-28 | d7-NMe-FOSE | 8.17 | R |
| EP1A-FORE-PART-01-091520 | L33784-28 | d9-NEt-FOSE | 10.5 | R |
| EP1A-FORE-WHO-01-091520 | L33784-29 | 13C2-PFTeDA | 34.2 | UJ |
| EP1A-FORE-WHO-01-091520 | L33784-29 | D3-N-MeFOSA | 22.4 | UJ |
| EP1A-FORE-WHO-01-091520 | L33784-29 | D5-N-EtFOSA | 18.6 | R |
| EP1A-FORE-WHO-01-091520 | L33784-29 | d7-NMe-FOSE | 12.1 | R |
| EP1A-FORE-WHO-01-091520 | L33784-29 | d9-NEt-FOSE | 3.92 | R |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | 13C2-PFTeDA | 25.8 | J+ |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | D3-N-MeFOSA | 36.0 | UJ |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | D5-N-EtFOSA | 15.0 | R |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | d7-NMe-FOSE | 2.50 | R |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | d9-NEt-FOSE | 6.77 | R |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | 13C2-PFTeDA | 27.6 | J+ |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | 13C2-4:2 FTS | 211 | No Qualifier |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | D3-N-MeFOSA | 23.1 | UJ |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | D5-N-EtFOSA | 12.3 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | d7-NMe-FOSE | 1.36 | R |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | d9-NEt-FOSE | 4.47 | R |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | 13C3-HFPO-DA | 43.9 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detections associated with high recoveries were qualified as estimated biased high (J+).

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74153-102 (A)/ WG74153-103 (DUP WG74153-102) | 11Cl-PF3OUdS | 141/**162** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 5:3 FTCA | **172/168** | 50-150 | |
| | 9Cl-PF3ONS | 135/**155** | 50-150 | |
| | N-MeFOSE | **168/216** | 50-150 | |
| | ADONA | 139/**158** | 50-150 | Associated detects were qualified as estimated biased high (J+):<br><br>EP18-FORE-WHO-TWO-01-091520 (L33784-22) |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| BP1-FORE-FIL-ONE-01-092020 | L33784-12 | PFOA |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | PFTeDA |
| BP1-FORE-PART-ONE-01-092020 | L33784-13 | N-EtFOSE |
| BP1-FORE-FIL-TWO-01-092020 | L33784-14 | PFDoA |
| BP1-FORE-PART-TWO-01-092020 | L33784-15 | N-EtFOSE |
| BP1-FORE-WHO-THREE-01-091920 | L33784-16 | N-EtFOSE |
| RC21A-FORE-WHO-01-092120 | L33784-17 | N-EtFOSE |
| WL7-FORE-WHO-01-091720 | L33784-19 | PFTrDA |
| WL7-FORE-WHO-01-091720 | L33784-19 | 6:2 FTS |
| WL7-FORE-WHO-01-091720 | L33784-19 | N-EtFOSE |
| WL7-FORE-WHO-01-091720 | L33784-19 | NFDHA |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | PFTrDA |
| EP18-FORE-PART-ONE-01-091520 | L33784-20 | N-EtFOSE |
| EP15-FORE-WHO-THREE-01-091820 | L33784-23 | N-EtFOSE |
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | PFDoA |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP27C-FORE-FIL-ONE-01-091520 | L33784-24 | N-EtFOSE |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | PFTrDA |
| EP27C-FORE-PART-ONE-01-091520 | L33784-25 | N-EtFOSE |
| EP27C-FORE-WHO-TWO-01-091520 | L33784-26 | N-EtFOSE |
| EP1A-FORE-PART-01-091520 | L33784-28 | N-EtFOSE |
| EP1A-FORE-WHO-01-091520 | L33784-29 | N-EtFOSE |
| EP9A-FORE-FIL-ONE-01-091820 | L33784-30 | N-EtFOSE |
| EP9A-FORE-PART-ONE-01-091820 | L33784-31 | N-EtFOSE |

The laboratory was unable to report results for samples listed below as the labeled analog recoveries were less than 1% and the results are unquantifiable.

| Sample ID | Analytes |
|---|---|
| RC18A-FORE-WHO-01-092120 (L33784-18) | N-MeFOSE<br>N-EtFOSE |
| EP15-FORE-WHO-THREE-01-091820 (L33784-23) | N-MeFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist


Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560


*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74690 Report date (mm/dd/yyyy): 12/2/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/13/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☐ | ☒ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | Bottle labels did not match the COC for one sample for those reported in this SDG. The sample was logged in using the ID on the COC. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Forty results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS tissue/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | N-MeFOSE 196%/181%<br>5:3 FTCA 157%/177%<br>Associated results were nondetect and were acceptable without qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | 50% limit for PFAS (other/tissue) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Forty results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/13/2020

## Data Validation Qualifications for DPWG74690

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/25/20 4:40 | 3:3 FTCA | 0% | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| CCV 11/25/20 8:34 | 3:3 FTCA | 0.3% | 70-130% | Only PFOS dilutions reported from this CCV. No qualifiers. |
| | PFMPA | 0% | 70-130% | |
| | NFDHA | 149% | 70-130% | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74154-101 | PFUnA | 0.139 J | WL2-FORE-WHO-ONE-01-091720 (L33784-33)<br>WL2-FORE-WHO-TWO-01-091720 (L33784-34)<br>WL6-FORE-WHO-01-091720 (L33784-35)<br>WL5-FORE-WHO-TWO-01-091720 (L33784-36)<br>WL3-FORE-WHO-ONE-01-091720 (L33784-37)<br>WL5-FORE-WHO-ONE-01-091720 (L33784-38)<br>EP14-FORE-FIL-ONE-01-091820 (L33784-39)<br>EP14-FORE-PART-ONE-01-091820 (L33784-40)<br>EP14-FORE-FIL-TWO-01-091820 (L33784-41)<br>WL8A-FORE-WHO-ONE-01-091620 (L33784-44)<br>WL8A-FORE-WHO-TWO-01-091620 (L33784-45)<br>WL21-FORE-WHO-ONE-01-091620 (L33784-46)<br>WL14-FORE-WHO-ONE-01-091620 (L33784-48)<br>WL14-FORE-WHO-TWO-01-091620 (L33784-49)<br>WL15A-FORE-WHO-TWO-01-091620 (L33784-51) |
| | PFTrDA | 0.170 J | All samples:<br>EP9A-FORE-WHO-TWO-01-091820 (L33784-32)<br>WL2-FORE-WHO-ONE-01-091720 (L33784-33)<br>WL2-FORE-WHO-TWO-01-091720 (L33784-34)<br>WL6-FORE-WHO-01-091720 (L33784-35)<br>WL5-FORE-WHO-TWO-01-091720 (L33784-36)<br>WL3-FORE-WHO-ONE-01-091720 (L33784-37)<br>WL5-FORE-WHO-ONE-01-091720 (L33784-38)<br>EP14-FORE-FIL-ONE-01-091820 (L33784-39)<br>EP14-FORE-PART-ONE-01-091820 (L33784-40)<br>EP14-FORE-FIL-TWO-01-091820 (L33784-41)<br>EP14-FORE-PART-TWO-01-091820 (L33784-42)<br>WL3-FORE-WHO-01-091720 (L33784-43)<br>WL8A-FORE-WHO-ONE-01-091620 (L33784-44)<br>WL8A-FORE-WHO-TWO-01-091620 (L33784-45)<br>WL21-FORE-WHO-ONE-01-091620 (L33784-46)<br>WL21-FORE-WHO-TWO-01-091620 (L33784-47)<br>WL14-FORE-WHO-ONE-01-091620 (L33784-48)<br>WL14-FORE-WHO-TWO-01-091620 (L33784-49) |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| | | | WL15A-FORE-WHO-ONE-01-091620 (L33784-50)<br>WL15A-FORE-WHO-TWO-01-091620 (L33784-51) |
| | PFOS | 0.193 J | Sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Forty results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9A-FORE-WHO-TWO-01-091820 | L33784-32 | 13C5-PFPeA | 263 | No Qualifier |
| EP9A-FORE-WHO-TWO-01-091820 | L33784-32 | D5-EtFOSAA | 214 | No Qualifier |
| EP9A-FORE-WHO-TWO-01-091820 | L33784-32 | d7-NMe-FOSE | 8.28 | R |
| EP9A-FORE-WHO-TWO-01-091820 | L33784-32 | d9-NEt-FOSE | 32.4 | UJ |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | 13C2-PFTeDA | 31.8 | J+ |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | D3-N-MeFOSA | 32.0 | UJ |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | D5-N-EtFOSA | 20.8 | UJ |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | D5-EtFOSAA | 201 | J- |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | d7-NMe-FOSE | 6.77 | R |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | d9-NEt-FOSE | 5.87 | R |
| WL2-FORE-WHO-TWO-01-091720 | L33784-34 | D3-N-MeFOSA | 39.5 | UJ |
| WL2-FORE-WHO-TWO-01-091720 | L33784-34 | D5-N-EtFOSA | 35.1 | UJ |
| WL2-FORE-WHO-TWO-01-091720 | L33784-34 | D5-EtFOSAA | 46.3 | J+ |
| WL2-FORE-WHO-TWO-01-091720 | L33784-34 | d7-NMe-FOSE | 4.73 | R |
| WL2-FORE-WHO-TWO-01-091720 | L33784-34 | d9-NEt-FOSE | 18.5 | R |
| WL6-FORE-WHO-01-091720 | L33784-35 | 13C2-PFTeDA | 37.9 | J+ |
| WL6-FORE-WHO-01-091720 | L33784-35 | D3-N-MeFOSA | 32.9 | UJ |
| WL6-FORE-WHO-01-091720 | L33784-35 | D5-N-EtFOSA | 27.5 | UJ |
| WL6-FORE-WHO-01-091720 | L33784-35 | d7-NMe-FOSE | 23.6 | UJ |
| WL6-FORE-WHO-01-091720 | L33784-35 | d9-NEt-FOSE | 6.73 | R |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | 13C2-4:2 FTS | 212 | No Qualifier |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | D3-N-MeFOSA | 25.9 | UJ |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | D5-N-EtFOSA | 19.0 | R |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | D5-EtFOSAA | 238 | J- |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | d7-NMe-FOSE | 13.4 | R |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | d9-NEt-FOSE | 4.66 | R |
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | D3-N-MeFOSA | 38.7 | UJ |
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | D5-N-EtFOSA | 23.3 | UJ |
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | D5-EtFOSAA | 250 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | d7-NMe-FOSE | 9.41 | R |
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | d9-NEt-FOSE | 5.20 | R |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | 13C2-PFTeDA | 32.4 | J+ |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | D3-N-MeFOSA | 26.3 | UJ |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | D5-N-EtFOSA | 17.0 | R |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | D5-EtFOSAA | 219 | J- |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | d7-NMe-FOSE | 9.78 | R |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | d9-NEt-FOSE | 6.40 | R |
| EP14-FORE-FIL-ONE-01-091820 | L33784-39 | D5-N-EtFOSA | 41.4 | UJ |
| EP14-FORE-FIL-ONE-01-091820 | L33784-39 | d7-NMe-FOSE | 12.0 | R |
| EP14-FORE-FIL-ONE-01-091820 | L33784-39 | d9-NEt-FOSE | 25.1 | UJ |
| EP14-FORE-PART-ONE-01-091820 | L33784-40 | 13C2-PFTeDA | 32.3 | J+ |
| EP14-FORE-PART-ONE-01-091820 | L33784-40 | D3-N-MeFOSA | 23.5 | UJ |
| EP14-FORE-PART-ONE-01-091820 | L33784-40 | D5-N-EtFOSA | 14.8 | R |
| EP14-FORE-PART-ONE-01-091820 | L33784-40 | d7-NMe-FOSE | 6.88 | R |
| EP14-FORE-PART-ONE-01-091820 | L33784-40 | d9-NEt-FOSE | 4.90 | R |
| EP14-FORE-FIL-TWO-01-091820 | L33784-41 | D5-N-EtFOSA | 39.6 | UJ |
| EP14-FORE-FIL-TWO-01-091820 | L33784-41 | d7-NMe-FOSE | 15.7 | R |
| EP14-FORE-FIL-TWO-01-091820 | L33784-41 | d9-NEt-FOSE | 24.6 | UJ |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | 13C2-4:2 FTS | 209 | No Qualifier |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | D3-N-MeFOSA | 49.0 | UJ |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | D5-N-EtFOSA | 22.1 | UJ |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | D5-EtFOSAA | 208 | J- |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | d7-NMe-FOSE | 4.38 | R |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | d9-NEt-FOSE | 14.6 | R |
| EP14-FORE-PART-TWO-01-091820 | L33784-42 | 13C3-HFPO-DA | 34.5 | UJ |
| WL3-FORE-WHO-01-091720 | L33784-43 | 13C2-PFTeDA | 42.6 | J+ |
| WL3-FORE-WHO-01-091720 | L33784-43 | D3-N-MeFOSA | 34.2 | UJ |
| WL3-FORE-WHO-01-091720 | L33784-43 | D5-N-EtFOSA | 19.1 | R |
| WL3-FORE-WHO-01-091720 | L33784-43 | D5-EtFOSAA | 202 | J- |
| WL3-FORE-WHO-01-091720 | L33784-43 | d7-NMe-FOSE | 5.40 | R |
| WL3-FORE-WHO-01-091720 | L33784-43 | d9-NEt-FOSE | 8.07 | R |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | 13C2-PFTeDA | 25.7 | J+ |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | D3-N-MeFOSA | 25.8 | UJ |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | D5-N-EtFOSA | 22.4 | UJ |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | d7-NMe-FOSE | 9.63 | R |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | d9-NEt-FOSE | 4.98 | R |
| WL8A-FORE-WHO-TWO-01-091620 | L33784-45 | D3-N-MeFOSA | 43.1 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL8A-FORE-WHO-TWO-01-091620 | L33784-45 | D5-N-EtFOSA | 30.6 | UJ |
| WL8A-FORE-WHO-TWO-01-091620 | L33784-45 | d7-NMe-FOSE | 8.54 | R |
| WL8A-FORE-WHO-TWO-01-091620 | L33784-45 | d9-NEt-FOSE | 10.9 | R |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | 13C2-4:2 FTS | 255 | No Qualifier |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | D3-N-MeFOSA | 36.8 | UJ |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | D5-N-EtFOSA | 22.2 | UJ |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | d7-NMe-FOSE | 3.23 | R |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | d9-NEt-FOSE | 9.30 | R |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | 13C2-PFTeDA | 29.1 | J+ |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | D3-N-MeFOSA | 36.1 | UJ |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | D5-N-EtFOSA | 16.4 | R |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | D5-EtFOSAA | 216 | J- |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | d7-NMe-FOSE | 2.65 | R |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | d9-NEt-FOSE | 10.4 | R |
| WL14-FORE-WHO-ONE-01-091620 | L33784-48 | D3-N-MeFOSA | 40.0 | UJ |
| WL14-FORE-WHO-ONE-01-091620 | L33784-48 | D5-N-EtFOSA | 29.1 | UJ |
| WL14-FORE-WHO-ONE-01-091620 | L33784-48 | d7-NMe-FOSE | 3.65 | R |
| WL14-FORE-WHO-ONE-01-091620 | L33784-48 | d9-NEt-FOSE | 12.4 | R |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | D3-N-MeFOSA | 40.3 | UJ |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | D5-N-EtFOSA | 23.8 | UJ |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | D5-EtFOSAA | 212 | J- |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | d7-NMe-FOSE | 3.12 | R |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | d9-NEt-FOSE | 14.9 | R |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | 13C2-PFTeDA | 21.8 | J+ |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | D3-N-MeFOSA | 32.8 | UJ |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | D5-N-EtFOSA | 20.1 | UJ |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | D5-EtFOSAA | 206 | J- |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | d7-NMe-FOSE | 11.8 | R |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | d9-NEt-FOSE | 6.32 | R |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | 13C2-PFTeDA | 31.4 | J+ |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | D3-N-MeFOSA | 24.7 | UJ |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | D5-N-EtFOSA | 16.0 | UJ |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | d7-NMe-FOSE | 9.19 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | d9-NEt-FOSE | 5.47 | R |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | PFTeDA |
| WL2-FORE-WHO-ONE-01-091720 | L33784-33 | N-EtFOSE |
| WL6-FORE-WHO-01-091720 | L33784-35 | N-EtFOSE |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | PFHxS |
| WL5-FORE-WHO-TWO-01-091720 | L33784-36 | N-EtFOSE |
| WL3-FORE-WHO-ONE-01-091720 | L33784-37 | N-EtFOSE |
| WL5-FORE-WHO-ONE-01-091720 | L33784-38 | N-EtFOSE |
| WL3-FORE-WHO-01-091720 | L33784-43 | PFHxS |
| WL3-FORE-WHO-01-091720 | L33784-43 | N-EtFOSE |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | PFTrDA |
| WL8A-FORE-WHO-ONE-01-091620 | L33784-44 | N-EtFOSE |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | PFDoA |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | PFTeDA |
| WL21-FORE-WHO-ONE-01-091620 | L33784-46 | N-EtFOSE |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | PFTeDA |
| WL21-FORE-WHO-TWO-01-091620 | L33784-47 | N-EtFOSE |
| WL14-FORE-WHO-TWO-01-091620 | L33784-49 | PFTeDA |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | PFDoA |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | PFTeDA |
| WL15A-FORE-WHO-ONE-01-091620 | L33784-50 | N-EtFOSE |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | PFTeDA |
| WL15A-FORE-WHO-TWO-01-091620 | L33784-51 | N-EtFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74712 Report date (mm/dd/yyyy): 12/7/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/13/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. A trip blank was received, but was not listed on the COC. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip Blank: 812443399253 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. One result was rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**One result was rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/13/2020

## Data Validation Qualifications for DPWG74712

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| VB3-FOAM-01-091620 | L33798-4 | 13C5-PFPeA | 45.6 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C5-PFHxA | 48 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C4-PFHpA | 49 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C8-PFOA | 44.7 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C9-PFNA | 45.8 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C6-PFDA | 40.5 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C7-PFUnA | 39 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C2-PFDoA | 30 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C2-PFTeDA | 17.4 | R |
| VB3-FOAM-01-091620 | L33798-4 | 13C3-PFBS | 44.9 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | 13C3-PFHxS | 44.9 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C8-PFOS | 38 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | 13C2-6:2 FTS | 31.7 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | 13C2-8:2 FTS | 30.6 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | 13C8-PFOSA | 43.2 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | D3-N-MeFOSA | 36.9 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | D5-N-EtFOSA | 32.6 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | D3-MeFOSAA | 27.9 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | D5-EtFOSAA | 41.3 | J+ |
| VB3-FOAM-01-091620 | L33798-4 | d7-NMe-FOSE | 35.6 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | d9-NEt-FOSE | 33.6 | UJ |
| VB3-FOAM-01-091620 | L33798-4 | 13C3-HFPO-DA | 40.5 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| VB3-FOAM-01-091620 | L33798-4 | EtFOSAA |

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74760 Report date (mm/dd/yyyy): 12/22/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/10/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. A trip blank was received, but was not listed on the COC. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Case narrative indicates samples were received at greater than 4 °C. Sample receipt was acceptable since they were received within the MPCA requirement of <6 °C. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL, ICV and CCV reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip Blank: 812443399253 (L33798-1) reported in SDG DPWG74712 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | No detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Thrity-one results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Thirty-one results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/10/2021

## Data Validation Qualifications for DPWG74760

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG74157-101 | PFBA | 0.653 J | EP27C-BOTF-WHO-01-091520 (L33786-1)<br>EP1A-BOTF-WHO-01-091520 (L33786-2)<br>RC7-BOTF-WHO-01-091820 (L33786-3)<br>RC22-BOTF-WHO-01-091820 (L33786-4)<br>RC18-BOTF-WHO-01-091920 (L33786-5)<br>RC18A-CRAY-01-092120 (L33788-1)<br>EP26A-CRAY-THREE-01-091720 (L33788-2)<br>RC3A-CRAY-01-092020 (L33788-3)<br>RC4A-CRAY-01-092520 (L33788-4)<br>RC23-CRAY-FOUR-01-092420 (L33788-5)<br>RC23-CRAY-ONE-01-092520 (L33788-6)<br>RC23-CRAY-TWO-01-092520 (L33788-7)<br>RC23-CRAY-THREE-01-092520 (L33788-8)<br>RC14-CRAY-01-092120 (L33788-10 i)<br>RC21A-CRAY-TWO-01-092120 (L33788-11)<br>RC21-CRAY-ONE-01-092120 (L33788-12)<br>RC21-CRAY-TWO-01-092120 (L33788-13)<br>RC21-CRAY-THREE-01-092120 (L33788-14)<br>RC21B-CRAY-01-092120 (L33788-15) |
| | PFUnA | 0.118 J | EP27C-BOTF-WHO-01-091520 (L33786-1)<br>EP1A-BOTF-WHO-01-091520 (L33786-2)<br>RC18-BOTF-WHO-01-091920 (L33786-5)<br>RC18A-CRAY-01-092120 (L33788-1)<br>EP26A-CRAY-THREE-01-091720 (L33788-2)<br>RC3A-CRAY-01-092020 (L33788-3)<br>RC4A-CRAY-01-092520 (L33788-4)<br>RC23-CRAY-TWO-01-092520 (L33788-7)<br>RC23-CRAY-COMP-01-092520 (L33788-9)<br>RC14-CRAY-01-092120 (L33788-10 i)<br>RC21A-CRAY-TWO-01-092120 (L33788-11)<br>RC21-CRAY-ONE-01-092120 (L33788-12)<br>RC21-CRAY-TWO-01-092120 (L33788-13)<br>RC21-CRAY-THREE-01-092120 (L33788-14)<br>RC21B-CRAY-01-092120 (L33788-15) |
| | PFTrDA | 0.153 J | EP1A-BOTF-WHO-01-091520 (L33786-2)<br>RC7-BOTF-WHO-01-091820 (L33786-3)<br>RC22-BOTF-WHO-01-091820 (L33786-4)<br>RC18-BOTF-WHO-01-091920 (L33786-5)<br>RC18A-CRAY-01-092120 (L33788-1)<br>RC3A-CRAY-01-092020 (L33788-3)<br>RC4A-CRAY-01-092520 (L33788-4)<br>RC23-CRAY-FOUR-01-092420 (L33788-5)<br>RC23-CRAY-ONE-01-092520 (L33788-6)<br>RC23-CRAY-TWO-01-092520 (L33788-7)<br>RC23-CRAY-THREE-01-092520 (L33788-8) |

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| | | | RC23-CRAY-COMP-01-092520 (L33788-9)<br>RC14-CRAY-01-092120 (L33788-10 i)<br>RC21A-CRAY-TWO-01-092120 (L33788-11)<br>RC21-CRAY-ONE-01-092120 (L33788-12)<br>RC21-CRAY-TWO-01-092120 (L33788-13)<br>RC21-CRAY-THREE-01-092120 (L33788-14)<br>RC21B-CRAY-01-092120 (L33788-15) |
| | PFOS | 0.153 J | Samples concentrations were greater than 10 times the blank concentration. No qualifiers. |
| | PFOSA | 0.109 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Thirty-one results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27C-BOTF-WHO-01-091520 | L33786-1 | 13C2-PFTeDA | 34.2 | UJ |
| EP27C-BOTF-WHO-01-091520 | L33786-1 | D3-N-MeFOSA | 24.1 | UJ |
| EP27C-BOTF-WHO-01-091520 | L33786-1 | D5-N-EtFOSA | 18.2 | UJ |
| EP27C-BOTF-WHO-01-091520 | L33786-1 | d7-NMe-FOSE | 1.56 | R |
| EP27C-BOTF-WHO-01-091520 | L33786-1 | d9-NEt-FOSE | 7.16 | R |
| EP27C-BOTF-WHO-01-091520 | L33786-1 | 13C3-HFPO-DA | 39.8 | UJ |
| EP1A-BOTF-WHO-01-091520 | L33786-2 | 13C2-PFTeDA | 43.3 | UJ |
| EP1A-BOTF-WHO-01-091520 | L33786-2 | D3-N-MeFOSA | 29.5 | UJ |
| EP1A-BOTF-WHO-01-091520 | L33786-2 | D5-N-EtFOSA | 17.7 | R |
| EP1A-BOTF-WHO-01-091520 | L33786-2 | d7-NMe-FOSE | 4.35 | R |
| EP1A-BOTF-WHO-01-091520 | L33786-2 | d9-NEt-FOSE | 7.87 | R |
| RC7-BOTF-WHO-01-091820 | L33786-3 | 13C2-PFTeDA | 38.3 | J+ |
| RC7-BOTF-WHO-01-091820 | L33786-3 | D3-N-MeFOSA | 29.3 | UJ |
| RC7-BOTF-WHO-01-091820 | L33786-3 | D5-N-EtFOSA | 21.6 | UJ |
| RC7-BOTF-WHO-01-091820 | L33786-3 | d7-NMe-FOSE | 20.2 | UJ |
| RC7-BOTF-WHO-01-091820 | L33786-3 | d9-NEt-FOSE | 7.21 | R |
| RC7-BOTF-WHO-01-091820 | L33786-3 | 13C3-HFPO-DA | 26.9 | UJ |
| RC22-BOTF-WHO-01-091820 | L33786-4 | 13C2-PFTeDA | 38.4 | J+ |
| RC22-BOTF-WHO-01-091820 | L33786-4 | D3-N-MeFOSA | 31.3 | UJ |
| RC22-BOTF-WHO-01-091820 | L33786-4 | D5-N-EtFOSA | 21.9 | UJ |
| RC22-BOTF-WHO-01-091820 | L33786-4 | d7-NMe-FOSE | 17.7 | R |
| RC22-BOTF-WHO-01-091820 | L33786-4 | d9-NEt-FOSE | 8.53 | R |
| RC22-BOTF-WHO-01-091820 | L33786-4 | 13C3-HFPO-DA | 37.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18-BOTF-WHO-01-091920 | L33786-5 | D3-N-MeFOSA | 41.0 | UJ |
| RC18-BOTF-WHO-01-091920 | L33786-5 | D5-N-EtFOSA | 29.0 | UJ |
| RC18-BOTF-WHO-01-091920 | L33786-5 | d7-NMe-FOSE | 7.01 | R |
| RC18-BOTF-WHO-01-091920 | L33786-5 | d9-NEt-FOSE | 13.0 | R |
| RC18-BOTF-WHO-01-091920 | L33786-5 | 13C3-HFPO-DA | 40.8 | UJ |
| RC18A-CRAY-01-092120 | L33788-1 | D3-N-MeFOSA | 40.3 | UJ |
| RC18A-CRAY-01-092120 | L33788-1 | D5-N-EtFOSA | 11.8 | R |
| RC18A-CRAY-01-092120 | L33788-1 | d7-NMe-FOSE | 8.58 | R |
| RC18A-CRAY-01-092120 | L33788-1 | d9-NEt-FOSE | 7.12 | R |
| EP26A-CRAY-THREE-01-091720 | L33788-2 | D3-N-MeFOSA | 40.1 | UJ |
| EP26A-CRAY-THREE-01-091720 | L33788-2 | D5-N-EtFOSA | 48.1 | UJ |
| EP26A-CRAY-THREE-01-091720 | L33788-2 | d7-NMe-FOSE | 24.6 | UJ |
| EP26A-CRAY-THREE-01-091720 | L33788-2 | d9-NEt-FOSE | 9.88 | R |
| RC3A-CRAY-01-092020 | L33788-3 | d9-NEt-FOSE | 19.9 | R |
| RC4A-CRAY-01-092520 | L33788-4 | 13C4-PFHpA | 31.7 | J+ |
| RC4A-CRAY-01-092520 | L33788-4 | D3-N-MeFOSA | 36.9 | UJ |
| RC4A-CRAY-01-092520 | L33788-4 | D5-N-EtFOSA | 43.2 | UJ |
| RC4A-CRAY-01-092520 | L33788-4 | d7-NMe-FOSE | 46.8 | UJ |
| RC4A-CRAY-01-092520 | L33788-4 | d9-NEt-FOSE | 9.59 | R |
| RC23-CRAY-FOUR-01-092420 | L33788-5 | D3-N-MeFOSA | 47.9 | UJ |
| RC23-CRAY-FOUR-01-092420 | L33788-5 | d7-NMe-FOSE | 46.7 | UJ |
| RC23-CRAY-FOUR-01-092420 | L33788-5 | d9-NEt-FOSE | 16.7 | R |
| RC23-CRAY-ONE-01-092520 | L33788-6 | D3-N-MeFOSA | 44.3 | UJ |
| RC23-CRAY-ONE-01-092520 | L33788-6 | d9-NEt-FOSE | 16.9 | R |
| RC23-CRAY-TWO-01-092520 | L33788-7 | D3-N-MeFOSA | 49.2 | UJ |
| RC23-CRAY-TWO-01-092520 | L33788-7 | d7-NMe-FOSE | 47.7 | UJ |
| RC23-CRAY-TWO-01-092520 | L33788-7 | d9-NEt-FOSE | 20.1 | UJ |
| RC23-CRAY-THREE-01-092520 | L33788-8 | D3-N-MeFOSA | 46.1 | UJ |
| RC23-CRAY-THREE-01-092520 | L33788-8 | d7-NMe-FOSE | 46.0 | UJ |
| RC23-CRAY-THREE-01-092520 | L33788-8 | d9-NEt-FOSE | 15.9 | R |
| RC23-CRAY-COMP-01-092520 | L33788-9 | D3-N-MeFOSA | 40.4 | UJ |
| RC23-CRAY-COMP-01-092520 | L33788-9 | D5-N-EtFOSA | 13.0 | R |
| RC23-CRAY-COMP-01-092520 | L33788-9 | d7-NMe-FOSE | 8.49 | R |
| RC23-CRAY-COMP-01-092520 | L33788-9 | d9-NEt-FOSE | 9.64 | R |
| RC14-CRAY-01-092120 | L33788-10 i | D3-N-MeFOSA | 42.9 | UJ |
| RC14-CRAY-01-092120 | L33788-10 i | D5-N-EtFOSA | 11.2 | R |
| RC14-CRAY-01-092120 | L33788-10 i | d7-NMe-FOSE | 25.6 | UJ |
| RC14-CRAY-01-092120 | L33788-10 i | d9-NEt-FOSE | 9.07 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC21A-CRAY-TWO-01-092120 | L33788-11 | D3-N-MeFOSA | 49.6 | UJ |
| RC21A-CRAY-TWO-01-092120 | L33788-11 | D5-N-EtFOSA | 16.8 | R |
| RC21A-CRAY-TWO-01-092120 | L33788-11 | d7-NMe-FOSE | 25.3 | UJ |
| RC21A-CRAY-TWO-01-092120 | L33788-11 | d9-NEt-FOSE | 13.9 | R |
| RC21-CRAY-ONE-01-092120 | L33788-12 | d9-NEt-FOSE | 15.0 | R |
| RC21-CRAY-TWO-01-092120 | L33788-13 | D3-N-MeFOSA | 48.8 | UJ |
| RC21-CRAY-TWO-01-092120 | L33788-13 | D5-N-EtFOSA | 21.9 | UJ |
| RC21-CRAY-TWO-01-092120 | L33788-13 | d7-NMe-FOSE | 30.5 | UJ |
| RC21-CRAY-TWO-01-092120 | L33788-13 | d9-NEt-FOSE | 15.4 | R |
| RC21-CRAY-THREE-01-092120 | L33788-14 | D3-N-MeFOSA | 12.2 | R |
| RC21-CRAY-THREE-01-092120 | L33788-14 | d7-NMe-FOSE | 41.2 | UJ |
| RC21-CRAY-THREE-01-092120 | L33788-14 | d9-NEt-FOSE | 11.9 | R |
| RC21B-CRAY-01-092120 | L33788-15 | 13C4-PFBA | 397 | J- |
| RC21B-CRAY-01-092120 | L33788-15 | D3-N-MeFOSA | 47.9 | UJ |
| RC21B-CRAY-01-092120 | L33788-15 | D5-N-EtFOSA | 21.1 | UJ |
| RC21B-CRAY-01-092120 | L33788-15 | d9-NEt-FOSE | 14.4 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with high recoveries were nondetect and were acceptable without qualification.

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74157-102 (A)/ WG74157-103 (DUP WG74157-102) | 5:3 FTCA | **173/174** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 11Cl-PF3OUdS | 140/**157** | 50-150 | |
| | 9Cl-PF3ONS | 136/**159** | 50-150 | |
| | ADONA | 137/**159** | 50-150 | |
| | N-MeFOSE | **162/163** | 50-150 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP1A-BOTF-WHO-01-091520 | L33786-2 | N-EtFOSE |
| RC7-BOTF-WHO-01-091820 | L33786-3 | N-EtFOSE |
| RC22-BOTF-WHO-01-091820 | L33786-4 | N-EtFOSE |
| RC18A-CRAY-01-092120 | L33788-1 | N-EtFOSE |
| RC4A-CRAY-01-092520 | L33788-4 | MeFOSAA |
| RC4A-CRAY-01-092520 | L33788-4 | N-EtFOSE |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC23-CRAY-FOUR-01-092420 | L33788-5 | PFNA |
| RC23-CRAY-TWO-01-092520 | L33788-7 | PFNA |
| RC23-CRAY-COMP-01-092520 | L33788-9 | N-EtFOSE |
| RC21A-CRAY-TWO-01-092120 | L33788-11 | PFBS |
| RC21-CRAY-TWO-01-092120 | L33788-13 | PFHxS |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74771 Report date (mm/dd/yyyy): 12/27/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/16/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | Case narrative indicates samples were received at greater than 4 °C. Sample receipt was acceptable since they were received within the MPCA requirement of <6 °C. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Sample WL15A-CRAY-EATEN-01-091620 (L33788-37) was not listed on the COC as it was a crayfish pulled from the fish sample WL15A-PRED-WHO-THREE-01-091620 that was reported in SDG DPWG74677 (L33776-49).<br><br>Descrepancies between the sample ID on the COC and sample labels were noted for 3 samples. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br><br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Login report indicates a trip blank L33798-1 was associated with the samples collected (reported in SDG DPWG74712) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than $1/10^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Forty results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Forty results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/16/2021

## Data Validation Qualifications for DPWG74771

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 26-Nov-2020 11:59 | NFDHA | 147 | 70-130 | Only PFOS and PFOSA dilutions reported from 11/26/20 analyses. No qualifiers. |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74155-101 | PFBA | 0.508 J | All results were within 10 times the blank concentration and qualified B. |
| | PFUnA | 0.105 J | WL15A-FORE-WHO-THREE-01-091620 (L33784-52)<br>WL15-FORE-WHO-ONE-01-091620 (L33784-54)<br>WL15-FORE-WHO-TWO-01-091620 (L33784-55)<br>WL15-FORE-WHO-THREE-01-091620 (L33784-56)<br>RC16A-FORE-COMP-ONE-01-091920 (L33784-63)<br>RC16A-FORE-COMP-TWO-01-091920 (L33784-64)<br>RC17-FORE-COMP-ONE-01-091920 (L33784-65)<br>RC17-FORE-COMP-TWO-01-091920 (L33784-66)<br>RC18-FORE-COMP-01-091920 (L33784-67)<br>EP15-FORE-WHO-ONE-01-091820 (L33784-70)<br>WL15A-CRAY-EATEN-01-091620 (L33788-37) |
| | PFOS | 0.153 J | Sample concentrations were greater than 10 times the blank concentration. No qualifiers. |
| | PFOSA | 0.109 J | WL15A-FORE-WHO-THREE-01-091620 (L33784-52)<br>WL15A-FORE-WHO-FOUR-01-091620 (L33784-53)<br>WL15-FORE-WHO-ONE-01-091620 (L33784-54)<br>WL15-FORE-WHO-TWO-01-091620 (L33784-55)<br>WL15-FORE-WHO-THREE-01-091620 (L33784-56)<br>RC16A-FORE-COMP-ONE-01-091920 (L33784-63)<br>EP15-FORE-WHO-ONE-01-091820 (L33784-70)<br>WL15A-CRAY-EATEN-01-091620 (L33788-37) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Forty results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | 13C2-PFTeDA | 30.9 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | D3-N-MeFOSA | 32.0 | UJ |
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | D5-N-EtFOSA | 24.8 | UJ |
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | D5-EtFOSAA | 218 | J- |
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | d7-NMe-FOSE | 2.69 | R |
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | d9-NEt-FOSE | 9.56 | R |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | 13C2-PFTeDA | 25.5 | J+ |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | D3-N-MeFOSA | 37.4 | UJ |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | D5-N-EtFOSA | 23.1 | UJ |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | D5-EtFOSAA | 224 | J- |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | d7-NMe-FOSE | 2.58 | R |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | d9-NEt-FOSE | 7.93 | R |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | 13C2-PFTeDA | 25.5 | UJ |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | D3-N-MeFOSA | 33.2 | UJ |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | D5-N-EtFOSA | 22.8 | UJ |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | D5-EtFOSAA | 211 | J- |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | d7-NMe-FOSE | 3.54 | R |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | d9-NEt-FOSE | 7.16 | R |
| WL15-FORE-WHO-TWO-01-091620 | L33784-55 | D3-N-MeFOSA | 36.2 | UJ |
| WL15-FORE-WHO-TWO-01-091620 | L33784-55 | D5-N-EtFOSA | 26.8 | UJ |
| WL15-FORE-WHO-TWO-01-091620 | L33784-55 | D5-EtFOSAA | 201 | J- |
| WL15-FORE-WHO-TWO-01-091620 | L33784-55 | d7-NMe-FOSE | 2.87 | R |
| WL15-FORE-WHO-TWO-01-091620 | L33784-55 | d9-NEt-FOSE | 14.0 | R |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | 13C2-PFTeDA | 30.8 | UJ |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | D3-N-MeFOSA | 35.8 | UJ |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | D5-N-EtFOSA | 23.7 | UJ |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | D5-EtFOSAA | 224 | J- |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | d7-NMe-FOSE | 5.13 | R |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | d9-NEt-FOSE | 8.47 | R |
| RC3A-FORE-COMP-ONE-01-091920 | L33784-57 | D5-EtFOSAA | 305 | J- |
| RC3A-FORE-COMP-ONE-01-091920 | L33784-57 | d7-NMe-FOSE | 6.02 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3A-FORE-COMP-ONE-01-091920 | L33784-57 | d9-NEt-FOSE | 23.6 | J+ |
| RC3A-FORE-COMP-ONE-01-091920 | L33784-57 | 13C3-HFPO-DA | 32.9 | UJ |
| RC4A-FORE-COMP-091820 | L33784-58 | 13C2-8:2 FTS | 201 | No Qualifier |
| RC4A-FORE-COMP-091820 | L33784-58 | D5-N-EtFOSA | 39.4 | J+ |
| RC4A-FORE-COMP-091820 | L33784-58 | D5-EtFOSAA | 359 | J- |
| RC4A-FORE-COMP-091820 | L33784-58 | d7-NMe-FOSE | 9.90 | R |
| RC4A-FORE-COMP-091820 | L33784-58 | d9-NEt-FOSE | 17.2 | R |
| RC4A-FORE-COMP-091820 | L33784-58 | 13C3-HFPO-DA | 23.2 | UJ |
| RC5-FORE-WHO-ONE-091820 | L33784-59 | D5-EtFOSAA | 454 | J- |
| RC5-FORE-WHO-ONE-091820 | L33784-59 | d7-NMe-FOSE | 8.05 | R |
| RC5-FORE-WHO-ONE-091820 | L33784-59 | d9-NEt-FOSE | 24.6 | UJ |
| RC5-FORE-WHO-ONE-091820 | L33784-59 | 13C3-HFPO-DA | 23.5 | UJ |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | 13C2-PFTeDA | 26.5 | J+ |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | D3-N-MeFOSA | 37.7 | UJ |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | D5-N-EtFOSA | 26.2 | UJ |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | D5-EtFOSAA | 320 | J- |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | d7-NMe-FOSE | 3.21 | R |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | d9-NEt-FOSE | 7.46 | R |
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | 13C3-HFPO-DA | 19.2 | R |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | 13C2-PFTeDA | 36.6 | J+ |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | 13C2-8:2 FTS | 203 | No Qualifier |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | 13C8-PFOSA | 219 | J- |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | D5-N-EtFOSA | 46.6 | UJ |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | D5-EtFOSAA | 297 | J- |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | d7-NMe-FOSE | 3.14 | R |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | d9-NEt-FOSE | 18.0 | R |
| RC22-FORE-WHO-091820 | L33784-62 | 13C2-4:2 FTS | 227 | No Qualifier |
| RC22-FORE-WHO-091820 | L33784-62 | 13C2-8:2 FTS | 202 | No Qualifier |
| RC22-FORE-WHO-091820 | L33784-62 | 13C8-PFOSA | 315 | J- |
| RC22-FORE-WHO-091820 | L33784-62 | D3-N-MeFOSA | 31.4 | UJ |
| RC22-FORE-WHO-091820 | L33784-62 | D5-N-EtFOSA | 44.8 | UJ |
| RC22-FORE-WHO-091820 | L33784-62 | D5-EtFOSAA | 462 | J- |
| RC22-FORE-WHO-091820 | L33784-62 | d7-NMe-FOSE | 27.1 | UJ |
| RC22-FORE-WHO-091820 | L33784-62 | d9-NEt-FOSE | 14.2 | R |
| RC22-FORE-WHO-091820 | L33784-62 | 13C3-HFPO-DA | 20.6 | UJ |
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | 13C2-4:2 FTS | 203 | No Qualifier |
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | D3-N-MeFOSA | 42.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | D5-N-EtFOSA | 28.9 | UJ |
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | d7-NMe-FOSE | 4.57 | R |
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | d9-NEt-FOSE | 13.2 | R |
| RC16A-FORE-COMP-ONE-01-091920 | L33784-63 | 13C3-HFPO-DA | 42.8 | UJ |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | 13C2-PFTeDA | 42.5 | J+ |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | 13C2-4:2 FTS | 201 | No Qualifier |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | D3-N-MeFOSA | 36.2 | UJ |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | D5-N-EtFOSA | 31.2 | UJ |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | d7-NMe-FOSE | 3.32 | R |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | d9-NEt-FOSE | 11.0 | R |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | D3-N-MeFOSA | 41.4 | UJ |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | D5-N-EtFOSA | 24.0 | UJ |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | d7-NMe-FOSE | 4.50 | R |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | d9-NEt-FOSE | 12.6 | R |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | 13C3-HFPO-DA | 41.1 | UJ |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | 13C2-PFTeDA | 41.4 | UJ |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | D3-N-MeFOSA | 30.8 | UJ |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | D5-N-EtFOSA | 18.5 | R |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | d7-NMe-FOSE | 3.20 | R |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | d9-NEt-FOSE | 7.27 | R |
| RC18-FORE-COMP-01-091920 | L33784-67 | 13C2-PFTeDA | 31.5 | J+ |
| RC18-FORE-COMP-01-091920 | L33784-67 | D3-N-MeFOSA | 33.9 | UJ |
| RC18-FORE-COMP-01-091920 | L33784-67 | D5-N-EtFOSA | 22.0 | UJ |
| RC18-FORE-COMP-01-091920 | L33784-67 | d7-NMe-FOSE | 3.10 | R |
| RC18-FORE-COMP-01-091920 | L33784-67 | d9-NEt-FOSE | 8.33 | R |
| RC18-FORE-COMP-01-091920 | L33784-67 | 13C3-HFPO-DA | 42.3 | UJ |
| EP17A-FORE-WHO-01-091920 | L33784-68 | 13C5-PFPeA | 275 | No Qualifier |
| EP17A-FORE-WHO-01-091920 | L33784-68 | 13C2-PFTeDA | 20.6 | UJ |
| EP17A-FORE-WHO-01-091920 | L33784-68 | 13C3-PFBS | 41.7 | UJ |
| EP17A-FORE-WHO-01-091920 | L33784-68 | D5-N-EtFOSA | 34.4 | UJ |
| EP17A-FORE-WHO-01-091920 | L33784-68 | D5-EtFOSAA | 268 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17A-FORE-WHO-01-091920 | L33784-68 | d7-NMe-FOSE | 3.42 | R |
| EP17A-FORE-WHO-01-091920 | L33784-68 | d9-NEt-FOSE | 12.7 | R |
| EP20-FORE-COMP-TWO-01-092020 | L33784-69 | D5-N-EtFOSA | 41.3 | UJ |
| EP20-FORE-COMP-TWO-01-092020 | L33784-69 | D5-EtFOSAA | 229 | J- |
| EP20-FORE-COMP-TWO-01-092020 | L33784-69 | d7-NMe-FOSE | 3.51 | R |
| EP20-FORE-COMP-TWO-01-092020 | L33784-69 | d9-NEt-FOSE | 24.0 | UJ |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | 13C2-PFTeDA | 48.0 | J+ |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | D3-N-MeFOSA | 26.3 | UJ |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | D5-N-EtFOSA | 17.2 | R |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | d7-NMe-FOSE | 1.17 | R |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | d9-NEt-FOSE | 6.62 | R |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | 13C5-PFPeA | 335 | No Qualifier |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | D3-N-MeFOSA | 23.8 | UJ |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | D5-N-EtFOSA | 7.18 | R |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | d7-NMe-FOSE | 5.78 | R |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | d9-NEt-FOSE | 3.50 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Results associated with high recoveries were nondetect and were acceptable without qualification.

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74155-102 (A)/ WG74155-103 (DUP WG74155-102) | 11Cl-PF3OUdS | 136/**177** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 5:3 FTCA | **171/168** | 50-150 | |
| | 9Cl-PF3ONS | 137/**183** | 50-150 | |
| | ADONA | 136/**175** | 50-150 | |
| | N-MeFOSE | **181/199** | 50-150 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | N-EtFOSE |
| WL15A-FORE-WHO-THREE-01-091620 | L33784-52 | NFDHA |
| WL15A-FORE-WHO-FOUR-01-091620 | L33784-53 | N-EtFOSE |
| WL15-FORE-WHO-ONE-01-091620 | L33784-54 | N-EtFOSE |
| WL15-FORE-WHO-THREE-01-091620 | L33784-56 | N-EtFOSE |
| RC3A-FORE-COMP-ONE-01-091920 | L33784-57 | N-EtFOSE |
| RC4A-FORE-COMP-091820 | L33784-58 | N-EtFOSE |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC7-FORE-COMP-ONE-01-092020 | L33784-60 | N-EtFOSE |
| RC22-FORE-COMP-TWO-091820 | L33784-61 | N-EtFOSE |
| RC22-FORE-WHO-091820 | L33784-62 | N-EtFOSE |
| RC16A-FORE-COMP-TWO-01-091920 | L33784-64 | N-EtFOSE |
| RC17-FORE-COMP-ONE-01-091920 | L33784-65 | 6:2 FTS |
| RC17-FORE-COMP-TWO-01-091920 | L33784-66 | N-EtFOSE |
| EP17A-FORE-WHO-01-091920 | L33784-68 | MeFOSAA |
| EP17A-FORE-WHO-01-091920 | L33784-68 | N-EtFOSE |
| EP15-FORE-WHO-ONE-01-091820 | L33784-70 | N-EtFOSE |
| WL15A-CRAY-EATEN-01-091620 | L33788-37 | N-EtFOSE |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74772 Report date (mm/dd/yyyy): 12/16/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/16/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | Case narrative indicates samples were received at greater than 4 °C. Sample receipt was acceptable since they were received within the MPCA requirement of <6 °C. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances and qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Equipment Blanks:<br>EQ-CATFOOD-01-100520 (L33799-1)<br>EQ-CHICKEN-01-100520 (L33799-2)<br>Login report indicates a trip blank L33798-1 was associated with the samples collected (reported in SDG DPWG74712) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | EQ-CATFOOD-01-100520 (L33799-1): PFOS detect attributable to laboratory contamination.<br>EQ-CHICKEN-01-100520 (L33799-2):<br>N-EtFOSE 2.11 J ng/g<br>Associated samples not provide. The equipment blank detection will be considered during ecological review of the results.<br>The trip blank results were nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Twenty-eight results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Twenty-eight results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/16/2021

## Data Validation Qualifications for DPWG74772

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 12-Nov-2020 14:32 | NFDHA | 144 | 70-130 | Associated results were nondetect. No qualifiers. |
| 12-Nov-2020 19:58 | NFDHA | 143 | 70-130 | |
| 12-Nov-2020 19:58 | NFDHA | 143 | 70-130 | |
| 01-Dec-2020 16:57 | NFDHA | 60.7 | 70-130 | Results qualified UJ:<br>WL16-AMPH-01-092320 (L33792-16 N) |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74160-101 | PFUnA | 0.118 J | WL10A-AMPH-01-092120 (L33792-14)<br>WL13-AMPH-ONE-01-092320 (L33792-15)<br>WL16-AMPH-01-092320 (L33792-16 N)<br>WL13-AMPH-TWO-01-092320 (L33808-4)<br>EP17C-SNAIL-ONE-01-092420 (L33808-5)<br>RC21A-CRAY-ONE-01-092120 (L33808-6)<br>RC21A-CRAY-THREE-01-092120 (L33808-7)<br>EP4-CRAY-ONE-01-092320 (L33808-8)<br>EP17A-FORE-COMP-ONE-01-091920 (L33808-9)<br>EP4-SNAIL-COMP-TWO-01-092420 (L33808-10)<br>RC3A-AMPH-COMP-TWO-01-092420 (L33808-11) |
| | PFTrDA | 0.122 J | WL10A-AMPH-01-092120 (L33792-14)<br>WL13-AMPH-ONE-01-092320 (L33792-15)<br>WL16-AMPH-01-092320 (L33792-16 N)<br>EP9A-PRED-WHO-FIVE-01-091820 (L33808-1)<br>BP1-PRED-PART-TWO-01-092020 (L33808-2)<br>EP9A-PRED-WHO-SIX-01-091820 (L33808-3 N2i)<br>WL13-AMPH-TWO-01-092320 (L33808-4)<br>RC21A-CRAY-ONE-01-092120 (L33808-6)<br>RC21A-CRAY-THREE-01-092120 (L33808-7)<br>EP17A-FORE-COMP-ONE-01-091920 (L33808-9)<br>RC3A-AMPH-COMP-TWO-01-092420 (L33808-11)<br>EP9A-PRED-PART-TWO-01-091820 (L33808-12) |
| | PFOS | 0.147 J | EQ-CATFOOD-01-100520 (L33799-1) |
| | PFOSA | 0.106 J | WL13-AMPH-ONE-01-092320 (L33792-15)<br>EP9A-PRED-WHO-FIVE-01-091820 (L33808-1)<br>BP1-PRED-PART-TWO-01-092020 (L33808-2)<br>EP9A-PRED-WHO-SIX-01-091820 (L33808-3 N2i)<br>WL13-AMPH-TWO-01-092320 (L33808-4)<br>EP9A-PRED-PART-TWO-01-091820 (L33808-12) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Twenty-eight results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL10A-AMPH-01-092120 | L33792-14 | D5-N-EtFOSA | 14.6 | R |
| WL10A-AMPH-01-092120 | L33792-14 | d7-NMe-FOSE | 39.7 | UJ |
| WL10A-AMPH-01-092120 | L33792-14 | d9-NEt-FOSE | 4.77 | R |
| WL13-AMPH-ONE-01-092320 | L33792-15 | 13C2-PFTeDA | 36.8 | UJ |
| WL13-AMPH-ONE-01-092320 | L33792-15 | 13C2-4:2 FTS | 224 | No Qualifier |
| WL13-AMPH-ONE-01-092320 | L33792-15 | 13C2-6:2 FTS | 213 | No Qualifier |
| WL13-AMPH-ONE-01-092320 | L33792-15 | 13C2-8:2 FTS | 227 | No Qualifier |
| WL13-AMPH-ONE-01-092320 | L33792-15 | D3-N-MeFOSA | 21.6 | UJ |
| WL13-AMPH-ONE-01-092320 | L33792-15 | D5-N-EtFOSA | 17.1 | R |
| WL13-AMPH-ONE-01-092320 | L33792-15 | d7-NMe-FOSE | 2.29 | R |
| WL13-AMPH-ONE-01-092320 | L33792-15 | d9-NEt-FOSE | 9.45 | R |
| WL16-AMPH-01-092320 | L33792-16 N | D5-N-EtFOSA | 17.1 | R |
| WL16-AMPH-01-092320 | L33792-16 N | d7-NMe-FOSE | 25.2 | UJ |
| WL16-AMPH-01-092320 | L33792-16 N | d9-NEt-FOSE | 6.64 | R |
| EQ-CATFOOD-01-100520 | L33799-1 | D5-N-EtFOSA | 14.8 | R |
| EQ-CATFOOD-01-100520 | L33799-1 | d7-NMe-FOSE | 26.7 | UJ |
| EQ-CATFOOD-01-100520 | L33799-1 | d9-NEt-FOSE | 17.9 | R |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | 13C2-PFTeDA | 18.5 | R |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | 13C2-4:2 FTS | 211 | No Qualifier |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | D3-N-MeFOSA | 29.8 | UJ |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | D5-N-EtFOSA | 14.9 | R |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | D5-EtFOSAA | 215 | J- |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | d7-NMe-FOSE | 1.36 | R |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | d9-NEt-FOSE | 6.78 | R |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | 13C3-HFPO-DA | 24.3 | UJ |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | 13C2-4:2 FTS | 219 | No Qualifier |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | D3-N-MeFOSA | 28.3 | UJ |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | D5-N-EtFOSA | 24.9 | UJ |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | d7-NMe-FOSE | 5.76 | R |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | d9-NEt-FOSE | 6.10 | R |
| EP9A-PRED-WHO-SIX-01-091820 | L33808-3 N2i | D5-N-EtFOSA | 46.6 | UJ |
| EP9A-PRED-WHO-SIX-01-091820 | L33808-3 N2i | d7-NMe-FOSE | 9.01 | R |
| EP9A-PRED-WHO-SIX-01-091820 | L33808-3 N2i | d9-NEt-FOSE | 25.4 | UJ |
| EP9A-PRED-WHO-SIX-01-091820 | L33808-3 N2i | 13C3-HFPO-DA | 29.5 | UJ |
| WL13-AMPH-TWO-01-092320 | L33808-4 | 13C2-4:2 FTS | 227 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL13-AMPH-TWO-01-092320 | L33808-4 | 13C2-6:2 FTS | 221 | No Qualifier |
| WL13-AMPH-TWO-01-092320 | L33808-4 | 13C2-8:2 FTS | 233 | No Qualifier |
| WL13-AMPH-TWO-01-092320 | L33808-4 | D3-N-MeFOSA | 32.2 | UJ |
| WL13-AMPH-TWO-01-092320 | L33808-4 | D5-N-EtFOSA | 43.9 | UJ |
| WL13-AMPH-TWO-01-092320 | L33808-4 | d7-NMe-FOSE | 11.3 | R |
| WL13-AMPH-TWO-01-092320 | L33808-4 | d9-NEt-FOSE | 16.0 | R |
| EP17C-SNAIL-ONE-01-092420 | L33808-5 | 13C2-PFTeDA | 41.4 | J+ |
| EP17C-SNAIL-ONE-01-092420 | L33808-5 | D3-N-MeFOSA | 28.6 | UJ |
| EP17C-SNAIL-ONE-01-092420 | L33808-5 | D5-N-EtFOSA | 33.5 | UJ |
| EP17C-SNAIL-ONE-01-092420 | L33808-5 | d7-NMe-FOSE | 29.3 | UJ |
| EP17C-SNAIL-ONE-01-092420 | L33808-5 | d9-NEt-FOSE | 10.5 | R |
| RC21A-CRAY-ONE-01-092120 | L33808-6 | 13C2-PFTeDA | 30.6 | J+ |
| RC21A-CRAY-ONE-01-092120 | L33808-6 | D3-N-MeFOSA | 26.2 | UJ |
| RC21A-CRAY-ONE-01-092120 | L33808-6 | D5-N-EtFOSA | 32.0 | UJ |
| RC21A-CRAY-ONE-01-092120 | L33808-6 | d7-NMe-FOSE | 32.7 | UJ |
| RC21A-CRAY-ONE-01-092120 | L33808-6 | d9-NEt-FOSE | 17.2 | R |
| RC21A-CRAY-THREE-01-092120 | L33808-7 | D3-N-MeFOSA | 46.5 | UJ |
| RC21A-CRAY-THREE-01-092120 | L33808-7 | D5-N-EtFOSA | 13.5 | R |
| RC21A-CRAY-THREE-01-092120 | L33808-7 | d7-NMe-FOSE | 25.2 | UJ |
| RC21A-CRAY-THREE-01-092120 | L33808-7 | d9-NEt-FOSE | 13.0 | R |
| EP4-CRAY-ONE-01-092320 | L33808-8 | 13C2-PFTeDA | 49.2 | UJ |
| EP4-CRAY-ONE-01-092320 | L33808-8 | D3-N-MeFOSA | 37.2 | UJ |
| EP4-CRAY-ONE-01-092320 | L33808-8 | D5-N-EtFOSA | 41.4 | UJ |
| EP4-CRAY-ONE-01-092320 | L33808-8 | d7-NMe-FOSE | 27.3 | UJ |
| EP4-CRAY-ONE-01-092320 | L33808-8 | d9-NEt-FOSE | 10.6 | R |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | 13C2-PFTeDA | 44.0 | J+ |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | 13C2-4:2 FTS | 201 | No Qualifier |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | D3-N-MeFOSA | 34.6 | UJ |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | D5-N-EtFOSA | 32.5 | UJ |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | d7-NMe-FOSE | 23.2 | UJ |
| EP17A-FORE-COMP-ONE-01-091920 | L33808-9 | d9-NEt-FOSE | 13.6 | R |
| EP4-SNAIL-COMP-TWO-01-092420 | L33808-10 | D3-N-MeFOSA | 32.2 | UJ |
| EP4-SNAIL-COMP-TWO-01-092420 | L33808-10 | D5-N-EtFOSA | 42.0 | UJ |
| EP4-SNAIL-COMP-TWO-01-092420 | L33808-10 | d7-NMe-FOSE | 38.6 | UJ |
| EP4-SNAIL-COMP-TWO-01-092420 | L33808-10 | d9-NEt-FOSE | 9.06 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | 13C2-4:2 FTS | 208 | No Qualifier |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | 13C2-6:2 FTS | 228 | No Qualifier |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | 13C2-8:2 FTS | 248 | No Qualifier |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | D3-N-MeFOSA | 46.1 | UJ |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | d7-NMe-FOSE | 10.6 | R |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | d9-NEt-FOSE | 14.8 | R |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | D5-N-EtFOSA | 39.6 | UJ |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | D5-EtFOSAA | 216 | J- |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | d7-NMe-FOSE | 9.65 | R |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | d9-NEt-FOSE | 38.9 | UJ |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | 13C3-HFPO-DA | 21.4 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Results associated with high recoveries were nondetect and were acceptable without qualification.

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74160-102 (A)/ WG74160-103 (DUP WG74160-102) | 5:3 FTCA | **160/161** | 50-150 | Associated results were nondetect. No qualifiers. |
| | N-MeFOSE | **183/206** | 50-150 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL10A-AMPH-01-092120 | L33792-14 | N-EtFOSE |
| WL13-AMPH-ONE-01-092320 | L33792-15 | PFNA |
| WL13-AMPH-ONE-01-092320 | L33792-15 | PFTrDA |
| WL16-AMPH-01-092320 | L33792-16 N | PFUnA |
| WL16-AMPH-01-092320 | L33792-16 N | N-EtFOSE |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | EtFOSAA |
| EP9A-PRED-WHO-FIVE-01-091820 | L33808-1 | N-EtFOSE |
| BP1-PRED-PART-TWO-01-092020 | L33808-2 | N-EtFOSE |
| WL13-AMPH-TWO-01-092320 | L33808-4 | EtFOSAA |
| EP4-SNAIL-COMP-TWO-01-092420 | L33808-10 | PFUnA |
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | PFNA |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3A-AMPH-COMP-TWO-01-092420 | L33808-11 | N-EtFOSE |
| EP9A-PRED-PART-TWO-01-091820 | L33808-12 | EtFOSAA |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74773 Report date (mm/dd/yyyy): 12/16/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/17/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Thirty-six results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for tissue (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Samples WL7-CRAY-TWO-01-092320, WL7-CRAY-FOUR-01-092320 and WL11-CRAY-01-092120 (SGS AXYS ID: L33788-28, -30 and -35, respectively) were instrumentally re-analyzed for possible sample carry-over impact on PFBA results. The re-analysis data confirmed that the analyte results for PFBA were affected and the results for PFBA were reported from the re-analysis data |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Thirty-six results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/17/2021

## Data Validation Qualifications for DPWG74773

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 08-Nov-2020 8:46 | NFDHA | 136 | 70-130 | Associated results were nondetect. No qualifiers. |
| 27-Nov-2020 18:58 | 3:3 FTCA | 0.6 | 70-130 | Only PFBA reported from 11/27/20. |
| | PFMPA | 0.0 | 70-130 | |
| 27-Nov-2020 22:53 | 11Cl-PF3OUdS | 0.0 | 70-130 | |
| | 3:3 FTCA | 0.1 | 70-130 | |
| | PFHpS | 0.0 | 70-130 | |
| | PFNA | 69.3 | 70-130 | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74158-101 | PFBA | 0.612 J | RC17-CRAY-ONE-01-091920 (L33788-16)<br>RC17A-CRAY-01-092120 (L33788-17)<br>EP17C-CRAY-01-092420 (L33788-18)<br>EP26A-CRAY-ONE-01-092520 (L33788-19)<br>EP26A-CRAY-TWO-01-092520 (L33788-20)<br>EP27B-CRAY-ONE-01-092420 (L33788-21)<br>EP27B-CRAY-TWO-01-092420 (L33788-22)<br>EP4-CRAY-TWO-01-092320 (L33788-23)<br>EP5-CRAY-ONE-01-092420 (L33788-24)<br>WL5-CRAY-01-091720 (L33788-25)<br>WL7-CRAY-ONE-01-092320 (L33788-27)<br>WL7-CRAY-TWO-01-092320 (L33788-28 i)<br>WL7-CRAY-FOUR-01-092320 (L33788-30 i)<br>WL8A-CRAY-01-092120 (L33788-31)<br>WL10-CRAY-ONE-01-092120 (L33788-32)<br>WL10-CRAY-TWO-01-092120 (L33788-33)<br>WL10-CRAY-THREE-01-092120 (L33788-34)<br>WL11-CRAY-01-092120 (L33788-35 i) |
| | PFUnA | 0.135 J | All PFUnA sample results were qualified B. |
| | PFTrDA | 0.198 J | RC17-CRAY-ONE-01-091920 (L33788-16)<br>RC17A-CRAY-01-092120 (L33788-17)<br>WL5-CRAY-01-091720 (L33788-25)<br>WL7-CRAY-ONE-01-092320 (L33788-27)<br>WL7-CRAY-TWO-01-092320 (L33788-28 i)<br>WL7-CRAY-THREE-01-092320 (L33788-29)<br>WL7-CRAY-FOUR-01-092320 (L33788-30 i)<br>WL8A-CRAY-01-092120 (L33788-31)<br>WL10-CRAY-THREE-01-092120 (L33788-34)<br>WL11-CRAY-01-092120 (L33788-35 i) |
| | PFOS | 0.142 J | Sample concentrations were greater than ten times the blank concentration. No qualifiers. |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| | PFOSA | 0.111 J | WL6-CRAY-01-092320 (L33788-26)<br>WL7-CRAY-ONE-01-092320 (L33788-27)<br>WL7-CRAY-TWO-01-092320 (L33788-28 i)<br>WL8A-CRAY-01-092120 (L33788-31)<br>WL10-CRAY-ONE-01-092120 (L33788-32)<br>WL10-CRAY-TWO-01-092120 (L33788-33)<br>WL10-CRAY-THREE-01-092120 (L33788-34)<br>WL11-CRAY-01-092120 (L33788-35 i) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Thirty-six results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC17-CRAY-ONE-01-091920 | L33788-16 | D3-N-MeFOSA | 47.0 | UJ |
| RC17-CRAY-ONE-01-091920 | L33788-16 | D5-N-EtFOSA | 12.8 | R |
| RC17-CRAY-ONE-01-091920 | L33788-16 | d7-NMe-FOSE | 7.85 | R |
| RC17-CRAY-ONE-01-091920 | L33788-16 | d9-NEt-FOSE | 14.2 | R |
| RC17A-CRAY-01-092120 | L33788-17 | D3-N-MeFOSA | 40.0 | UJ |
| RC17A-CRAY-01-092120 | L33788-17 | D5-N-EtFOSA | 8.95 | R |
| RC17A-CRAY-01-092120 | L33788-17 | d7-NMe-FOSE | 10.0 | R |
| RC17A-CRAY-01-092120 | L33788-17 | d9-NEt-FOSE | 7.54 | R |
| RC17A-CRAY-01-092120 | L33788-17 | 13C3-HFPO-DA | 18.1 | R |
| EP17C-CRAY-01-092420 | L33788-18 | D3-N-MeFOSA | 4.39 | R |
| EP17C-CRAY-01-092420 | L33788-18 | D5-N-EtFOSA | 4.99 | R |
| EP17C-CRAY-01-092420 | L33788-18 | d7-NMe-FOSE | 33.2 | UJ |
| EP17C-CRAY-01-092420 | L33788-18 | d9-NEt-FOSE | 12.8 | R |
| EP26A-CRAY-ONE-01-092520 | L33788-19 | D5-N-EtFOSA | 21.9 | UJ |
| EP26A-CRAY-ONE-01-092520 | L33788-19 | d7-NMe-FOSE | 17.4 | R |
| EP26A-CRAY-ONE-01-092520 | L33788-19 | d9-NEt-FOSE | 16.8 | R |
| EP26A-CRAY-TWO-01-092520 | L33788-20 | D3-N-MeFOSA | 31.3 | UJ |
| EP26A-CRAY-TWO-01-092520 | L33788-20 | D5-N-EtFOSA | 35.2 | UJ |
| EP26A-CRAY-TWO-01-092520 | L33788-20 | d7-NMe-FOSE | 24.6 | UJ |
| EP26A-CRAY-TWO-01-092520 | L33788-20 | d9-NEt-FOSE | 6.65 | R |
| EP27B-CRAY-ONE-01-092420 | L33788-21 | D3-N-MeFOSA | 47.5 | UJ |
| EP27B-CRAY-ONE-01-092420 | L33788-21 | d7-NMe-FOSE | 36.1 | UJ |
| EP27B-CRAY-ONE-01-092420 | L33788-21 | d9-NEt-FOSE | 12.3 | R |
| EP27B-CRAY-TWO-01-092420 | L33788-22 | D5-N-EtFOSA | 26.3 | UJ |
| EP27B-CRAY-TWO-01-092420 | L33788-22 | d7-NMe-FOSE | 16.5 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27B-CRAY-TWO-01-092420 | L33788-22 | d9-NEt-FOSE | 19.6 | R |
| EP4-CRAY-TWO-01-092320 | L33788-23 | 13C2-PFTeDA | 45.2 | UJ |
| EP4-CRAY-TWO-01-092320 | L33788-23 | D3-N-MeFOSA | 33.4 | UJ |
| EP4-CRAY-TWO-01-092320 | L33788-23 | D5-N-EtFOSA | 36.2 | UJ |
| EP4-CRAY-TWO-01-092320 | L33788-23 | d7-NMe-FOSE | 37.6 | UJ |
| EP4-CRAY-TWO-01-092320 | L33788-23 | d9-NEt-FOSE | 16.3 | R |
| EP5-CRAY-ONE-01-092420 | L33788-24 | D3-N-MeFOSA | 42.4 | UJ |
| EP5-CRAY-ONE-01-092420 | L33788-24 | d7-NMe-FOSE | 35.9 | UJ |
| EP5-CRAY-ONE-01-092420 | L33788-24 | d9-NEt-FOSE | 11.0 | R |
| WL5-CRAY-01-091720 | L33788-25 | 13C2-PFTeDA | 46.1 | J+ |
| WL5-CRAY-01-091720 | L33788-25 | D3-N-MeFOSA | 43.2 | UJ |
| WL5-CRAY-01-091720 | L33788-25 | D5-N-EtFOSA | 46.5 | UJ |
| WL5-CRAY-01-091720 | L33788-25 | d7-NMe-FOSE | 33.2 | UJ |
| WL5-CRAY-01-091720 | L33788-25 | d9-NEt-FOSE | 8.91 | R |
| WL6-CRAY-01-092320 | L33788-26 | D3-N-MeFOSA | 28.1 | UJ |
| WL6-CRAY-01-092320 | L33788-26 | D5-N-EtFOSA | 28.6 | UJ |
| WL6-CRAY-01-092320 | L33788-26 | d7-NMe-FOSE | 4.23 | R |
| WL6-CRAY-01-092320 | L33788-26 | d9-NEt-FOSE | 11.0 | R |
| WL7-CRAY-ONE-01-092320 | L33788-27 | D3-N-MeFOSA | 38.2 | UJ |
| WL7-CRAY-ONE-01-092320 | L33788-27 | D5-N-EtFOSA | 21.9 | UJ |
| WL7-CRAY-ONE-01-092320 | L33788-27 | d7-NMe-FOSE | 9.90 | R |
| WL7-CRAY-ONE-01-092320 | L33788-27 | d9-NEt-FOSE | 8.59 | R |
| WL7-CRAY-TWO-01-092320 | L33788-28 i | D3-N-MeFOSA | 42.6 | UJ |
| WL7-CRAY-TWO-01-092320 | L33788-28 i | d7-NMe-FOSE | 24.8 | UJ |
| WL7-CRAY-TWO-01-092320 | L33788-28 i | d9-NEt-FOSE | 9.36 | R |
| WL7-CRAY-THREE-01-092320 | L33788-29 | 13C5-PFPeA | 237 | No Qualifier |
| WL7-CRAY-THREE-01-092320 | L33788-29 | 13C2-PFTeDA | 44.1 | UJ |
| WL7-CRAY-THREE-01-092320 | L33788-29 | D3-N-MeFOSA | 36.6 | UJ |
| WL7-CRAY-THREE-01-092320 | L33788-29 | D5-N-EtFOSA | 36.0 | UJ |
| WL7-CRAY-THREE-01-092320 | L33788-29 | d7-NMe-FOSE | 15.0 | R |
| WL7-CRAY-THREE-01-092320 | L33788-29 | d9-NEt-FOSE | 9.21 | R |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | D3-N-MeFOSA | 25.8 | UJ |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | D5-N-EtFOSA | 20.1 | UJ |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | d7-NMe-FOSE | 4.68 | R |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | d9-NEt-FOSE | 6.86 | R |
| WL8A-CRAY-01-092120 | L33788-31 | D3-N-MeFOSA | 45.0 | UJ |
| WL8A-CRAY-01-092120 | L33788-31 | d7-NMe-FOSE | 10.6 | R |
| WL8A-CRAY-01-092120 | L33788-31 | d9-NEt-FOSE | 13.8 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL10-CRAY-ONE-01-092120 | L33788-32 | D3-N-MeFOSA | 41.7 | UJ |
| WL10-CRAY-ONE-01-092120 | L33788-32 | D5-N-EtFOSA | 45.0 | UJ |
| WL10-CRAY-ONE-01-092120 | L33788-32 | d7-NMe-FOSE | 31.8 | UJ |
| WL10-CRAY-ONE-01-092120 | L33788-32 | d9-NEt-FOSE | 12.3 | R |
| WL10-CRAY-ONE-01-092120 | L33788-32 | 13C3-HFPO-DA | 27.6 | UJ |
| WL10-CRAY-TWO-01-092120 | L33788-33 | D3-N-MeFOSA | 43.5 | UJ |
| WL10-CRAY-TWO-01-092120 | L33788-33 | D5-N-EtFOSA | 41.9 | UJ |
| WL10-CRAY-TWO-01-092120 | L33788-33 | d7-NMe-FOSE | 23.2 | UJ |
| WL10-CRAY-TWO-01-092120 | L33788-33 | d9-NEt-FOSE | 11.2 | R |
| WL10-CRAY-THREE-01-092120 | L33788-34 | D3-N-MeFOSA | 36.5 | UJ |
| WL10-CRAY-THREE-01-092120 | L33788-34 | D5-N-EtFOSA | 25.1 | UJ |
| WL10-CRAY-THREE-01-092120 | L33788-34 | d7-NMe-FOSE | 4.78 | R |
| WL10-CRAY-THREE-01-092120 | L33788-34 | d9-NEt-FOSE | 8.02 | R |
| WL10-CRAY-THREE-01-092120 | L33788-34 | 13C3-HFPO-DA | 32.5 | UJ |
| WL11-CRAY-01-092120 | L33788-35 i | D3-N-MeFOSA | 38.0 | UJ |
| WL11-CRAY-01-092120 | L33788-35 i | D5-N-EtFOSA | 39.5 | UJ |
| WL11-CRAY-01-092120 | L33788-35 i | d7-NMe-FOSE | 7.35 | R |
| WL11-CRAY-01-092120 | L33788-35 i | d9-NEt-FOSE | 17.1 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Results associated with high recoveries were nondetect and were acceptable without qualification.

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74158-102 (A)/ WG74158-103 (DUP WG74158-102) | N-MeFOSE | **156/160** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 5:3 FTCA | 149/**165** | 50-150 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC17-CRAY-ONE-01-091920 | L33788-16 | PFHxS |
| RC17A-CRAY-01-092120 | L33788-17 | N-EtFOSE |
| EP17C-CRAY-01-092420 | L33788-18 | PFTeDA |
| EP17C-CRAY-01-092420 | L33788-18 | PFPeS |
| EP17C-CRAY-01-092420 | L33788-18 | EtFOSAA |
| EP26A-CRAY-ONE-01-092520 | L33788-19 | PFBS |
| EP26A-CRAY-ONE-01-092520 | L33788-19 | PFHxS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP26A-CRAY-TWO-01-092520 | L33788-20 | N-EtFOSE |
| EP4-CRAY-TWO-01-092320 | L33788-23 | EtFOSAA |
| EP5-CRAY-ONE-01-092420 | L33788-24 | EtFOSAA |
| WL5-CRAY-01-091720 | L33788-25 | PFTeDA |
| WL5-CRAY-01-091720 | L33788-25 | N-EtFOSE |
| WL6-CRAY-01-092320 | L33788-26 | PFTeDA |
| WL7-CRAY-ONE-01-092320 | L33788-27 | EtFOSAA |
| WL7-CRAY-ONE-01-092320 | L33788-27 | N-EtFOSE |
| WL7-CRAY-THREE-01-092320 | L33788-29 | PFUnA |
| WL7-CRAY-THREE-01-092320 | L33788-29 | PFDoA |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | PFTeDA |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | EtFOSAA |
| WL7-CRAY-FOUR-01-092320 | L33788-30 i | N-EtFOSE |
| WL8A-CRAY-01-092120 | L33788-31 | PFTeDA |
| WL10-CRAY-TWO-01-092120 | L33788-33 | PFNA |
| WL10-CRAY-TWO-01-092120 | L33788-33 | PFDoA |
| WL10-CRAY-THREE-01-092120 | L33788-34 | N-EtFOSE |
| WL11-CRAY-01-092120 | L33788-35 i | PFTrDA |
| WL11-CRAY-01-092120 | L33788-35 i | EtFOSAA |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG74774 Report date (mm/dd/yyyy): 12/16/2020

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/17/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | Case narrative indicates samples were received at greater than 4 °C. Sample receipt was acceptable since they were received within the MPCA requirement of <6 °C. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The laboratory indicated one sample ID on the COC did not match the sample label. The sample was logged in per COC. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCV reviewed.<br>See data qualifications summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Thirty-three results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for tissue (other)<br>LCSD: See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Five samples were analyzed with a sample size less of than 2 grams resulting in elevated reporting limits. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Thirty-three results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/17/2021

## Data Validation Qualifications for DPWG74774

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 13-Nov-2020 12:14 | NFDHA | 144 | 70-130 | Associated results were nondetect. No qualifiers. |
| 23-Nov-2020 15:58 | 3:3 FTCA | 58.4 | 70-130 | Only PFOS reported from 11/23/20. |
| | NFDHA | 132 | 70-130 | |
| 23-Nov-2020 20:18 | 3:3 FTCA | 0.1 | 70-130 | |

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| WG74159-101 | PFUnA | 0.139 J | All PFUnA sample results were qualified B. |
| | PFTrDA | 0.198 J | RC3A-SNAIL-COMP-01-092420 (L33791-1)<br>RC4A-AMPH-COMP-01-091820 (L33792-1)<br>RC5-AMPH-ONE-01-091820 (L33792-2)<br>RC5-AMPH-TWO-01-092220 (L33792-3)<br>RC6A-AMPH-01-092020 (L33792-4)<br>RC3A-AMPH-COMP-ONE-01-091920 (L33792-5)<br>RC7-AMPH-01-092220 (L33792-6)<br>RC13-AMPH-01-092120 (L33792-7)<br>RC16A-AMPH-01-091920 (L33792-8)<br>RC17A-AMPH-01-092120 (L33792-9)<br>RC18-AMPH-01-091520 (L33792-10)<br>WL2-AMPH-01-092220 (L33792-12)<br>WL8A-AMPH-01-092520 (L33792-13) |
| | PFOS | 0.173 J | Sample concentrations were greater than ten times the blank concentration. No qualifiers. |
| | PFOSA | 0.101 J | RC17A-AMPH-01-092120 (L33792-9)<br>EP19-AMPH-01-091920 (L33792-11)<br>WL8A-AMPH-01-092520 (L33792-13) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Thirty-three results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL14-CRAY-01-092320 | L33788-36 | 13C2-PFTeDA | 44.5 | UJ |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | 13C2-PFTeDA | 32.6 | J+ |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | D3-N-MeFOSA | 36.0 | UJ |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | D5-N-EtFOSA | 32.8 | UJ |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | d7-NMe-FOSE | 19.2 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | d9-NEt-FOSE | 6.11 | R |
| RC23-SNAIL-COMP-01-092420 | L33791-2 | 13C2-PFTeDA | 39.3 | UJ |
| RC23-SNAIL-COMP-01-092420 | L33791-2 | d7-NMe-FOSE | 38.3 | UJ |
| RC23-SNAIL-COMP-01-092420 | L33791-2 | d9-NEt-FOSE | 8.05 | R |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | D3-N-MeFOSA | 34.8 | UJ |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | D5-N-EtFOSA | 46.8 | UJ |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | d7-NMe-FOSE | 38.4 | UJ |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | d9-NEt-FOSE | 11.2 | R |
| EP27B-SNAIL-TWO-01-092420 | L33791-4 | D3-N-MeFOSA | 38.1 | UJ |
| EP27B-SNAIL-TWO-01-092420 | L33791-4 | D5-N-EtFOSA | 33.1 | UJ |
| EP27B-SNAIL-TWO-01-092420 | L33791-4 | d7-NMe-FOSE | 25.2 | UJ |
| EP27B-SNAIL-TWO-01-092420 | L33791-4 | d9-NEt-FOSE | 12.2 | R |
| EP17C-SNAIL-TWO-01-092420 | L33791-5 | 13C2-PFTeDA | 23.8 | J+ |
| EP17C-SNAIL-TWO-01-092420 | L33791-5 | D3-N-MeFOSA | 23.8 | UJ |
| EP17C-SNAIL-TWO-01-092420 | L33791-5 | D5-N-EtFOSA | 27.6 | UJ |
| EP17C-SNAIL-TWO-01-092420 | L33791-5 | d7-NMe-FOSE | 24.0 | UJ |
| EP17C-SNAIL-TWO-01-092420 | L33791-5 | d9-NEt-FOSE | 7.46 | R |
| EP4-SNAIL-ONE-01-092420 | L33791-6 | D3-N-MeFOSA | 41.8 | UJ |
| EP4-SNAIL-ONE-01-092420 | L33791-6 | D5-N-EtFOSA | 45.8 | UJ |
| EP4-SNAIL-ONE-01-092420 | L33791-6 | d7-NMe-FOSE | 34.0 | UJ |
| EP4-SNAIL-ONE-01-092420 | L33791-6 | d9-NEt-FOSE | 17.1 | R |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | 13C2-PFTeDA | 46.6 | J+ |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | 13C2-4:2 FTS | 261 | No Qualifier |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | 13C2-6:2 FTS | 231 | No Qualifier |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | 13C2-8:2 FTS | 231 | No Qualifier |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | D3-N-MeFOSA | 10.1 | R |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | D5-N-EtFOSA | 27.5 | UJ |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | d7-NMe-FOSE | 5.62 | R |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | d9-NEt-FOSE | 5.12 | R |
| RC5-AMPH-ONE-01-091820 | L33792-2 | 13C2-PFTeDA | 29.5 | J+ |
| RC5-AMPH-ONE-01-091820 | L33792-2 | D3-N-MeFOSA | 30.7 | UJ |
| RC5-AMPH-ONE-01-091820 | L33792-2 | D5-N-EtFOSA | 19.5 | R |
| RC5-AMPH-ONE-01-091820 | L33792-2 | d7-NMe-FOSE | 20.3 | UJ |
| RC5-AMPH-ONE-01-091820 | L33792-2 | d9-NEt-FOSE | 6.44 | R |
| RC5-AMPH-TWO-01-092220 | L33792-3 | 13C2-PFTeDA | 27.4 | J+ |
| RC5-AMPH-TWO-01-092220 | L33792-3 | D3-N-MeFOSA | 24.5 | UJ |
| RC5-AMPH-TWO-01-092220 | L33792-3 | D5-N-EtFOSA | 23.9 | UJ |
| RC5-AMPH-TWO-01-092220 | L33792-3 | d7-NMe-FOSE | 22.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC5-AMPH-TWO-01-092220 | L33792-3 | d9-NEt-FOSE | 5.47 | R |
| RC6A-AMPH-01-092020 | L33792-4 | 13C2-PFTeDA | 20.2 | J+ |
| RC6A-AMPH-01-092020 | L33792-4 | D3-N-MeFOSA | 18.0 | R |
| RC6A-AMPH-01-092020 | L33792-4 | D5-N-EtFOSA | 18.1 | R |
| RC6A-AMPH-01-092020 | L33792-4 | d7-NMe-FOSE | 19.1 | R |
| RC6A-AMPH-01-092020 | L33792-4 | d9-NEt-FOSE | 4.85 | R |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | 13C2-PFTeDA | 45.4 | J+ |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | 13C2-8:2 FTS | 212 | No Qualifier |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | D3-N-MeFOSA | 17.7 | R |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | D5-N-EtFOSA | 7.62 | R |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | d7-NMe-FOSE | 3.94 | R |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | d9-NEt-FOSE | 5.45 | R |
| RC7-AMPH-01-092220 | L33792-6 | 13C2-PFTeDA | 44.6 | J+ |
| RC7-AMPH-01-092220 | L33792-6 | D3-N-MeFOSA | 42.1 | UJ |
| RC7-AMPH-01-092220 | L33792-6 | D5-N-EtFOSA | 37.6 | UJ |
| RC7-AMPH-01-092220 | L33792-6 | d7-NMe-FOSE | 37.3 | UJ |
| RC7-AMPH-01-092220 | L33792-6 | d9-NEt-FOSE | 12.1 | R |
| RC13-AMPH-01-092120 | L33792-7 | 13C2-PFTeDA | 18.5 | R |
| RC13-AMPH-01-092120 | L33792-7 | D3-N-MeFOSA | 17.9 | R |
| RC13-AMPH-01-092120 | L33792-7 | D5-N-EtFOSA | 14.4 | R |
| RC13-AMPH-01-092120 | L33792-7 | d7-NMe-FOSE | 15.1 | R |
| RC13-AMPH-01-092120 | L33792-7 | d9-NEt-FOSE | 3.04 | R |
| RC16A-AMPH-01-091920 | L33792-8 | 13C2-4:2 FTS | 206 | No Qualifier |
| RC16A-AMPH-01-091920 | L33792-8 | 13C2-8:2 FTS | 219 | No Qualifier |
| RC16A-AMPH-01-091920 | L33792-8 | D5-N-EtFOSA | 49.6 | UJ |
| RC16A-AMPH-01-091920 | L33792-8 | d7-NMe-FOSE | 25.9 | UJ |
| RC16A-AMPH-01-091920 | L33792-8 | d9-NEt-FOSE | 37.3 | UJ |
| RC17A-AMPH-01-092120 | L33792-9 | 13C2-4:2 FTS | 216 | No Qualifier |
| RC17A-AMPH-01-092120 | L33792-9 | D5-N-EtFOSA | 41.7 | UJ |
| RC17A-AMPH-01-092120 | L33792-9 | d9-NEt-FOSE | 23.0 | UJ |
| RC18-AMPH-01-091520 | L33792-10 | D5-N-EtFOSA | 38.8 | UJ |
| RC18-AMPH-01-091520 | L33792-10 | d9-NEt-FOSE | 19.6 | R |
| EP19-AMPH-01-091920 | L33792-11 | 13C2-4:2 FTS | 206 | No Qualifier |
| EP19-AMPH-01-091920 | L33792-11 | D5-N-EtFOSA | 39.2 | UJ |
| EP19-AMPH-01-091920 | L33792-11 | d9-NEt-FOSE | 22.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL2-AMPH-01-092220 | L33792-12 | D5-N-EtFOSA | 17.5 | R |
| WL2-AMPH-01-092220 | L33792-12 | d7-NMe-FOSE | 22.0 | UJ |
| WL2-AMPH-01-092220 | L33792-12 | d9-NEt-FOSE | 6.09 | R |
| WL8A-AMPH-01-092520 | L33792-13 | 13C2-4:2 FTS | 244 | No Qualifier |
| WL8A-AMPH-01-092520 | L33792-13 | 13C2-6:2 FTS | 248 | No Qualifier |
| WL8A-AMPH-01-092520 | L33792-13 | 13C2-8:2 FTS | 238 | No Qualifier |
| WL8A-AMPH-01-092520 | L33792-13 | D3-N-MeFOSA | 12.8 | R |
| WL8A-AMPH-01-092520 | L33792-13 | D5-N-EtFOSA | 26.0 | UJ |
| WL8A-AMPH-01-092520 | L33792-13 | d7-NMe-FOSE | 8.91 | R |
| WL8A-AMPH-01-092520 | L33792-13 | d9-NEt-FOSE | 14.8 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detections associated with high recoveries were qualified as estimated biased high (J+).

| LCS/LCSD IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG74159-102 (A)/ WG74159-103 (DUP WG74159-102) | N-MeFOSE | **262/173** | 50-150 | Associated results were nondetect. No qualifiers. |
| | 5:3 FTCA | 148/**162** | 50-150 | Associated samples detects were qualified as estimated biased high (J+):<br><br>RC5-AMPH-ONE-01-091820 (L33792-2)<br>RC13-AMPH-01-092120 (L33792-7) |

**Bold indicates an exceedance**

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detections associated with high RPDs were qualified as estimated (J).

| LCS/LCSD IDs | Compound | LCS/LCSD %RPD | RPD Limits | Qualifiers |
|---|---|---|---|---|
| WG74159-102 (A)/ WG74159-103 (DUP WG74159-102) | N-EtFOSE | **67.2** | 50 | Detects qualified as estimated (J):<br><br>RC3A-SNAIL-COMP-01-092420 (L33791-1)<br>RC23-SNAIL-COMP-01-092420 (L33791-2)<br>RC5-AMPH-ONE-01-091820 (L33792-2)<br>RC5-AMPH-TWO-01-092220 (L33792-3)<br>RC6A-AMPH-01-092020 (L33792-4)<br>RC3A-AMPH-COMP-ONE-01-091920 (L33792-5)<br>RC13-AMPH-01-092120 (L33792-7)<br>WL2-AMPH-01-092220 (L33792-12) |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria. These results were reported as maximum possible

concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL14-CRAY-01-092320 | L33788-36 | PFUnA |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | MeFOSAA |
| RC3A-SNAIL-COMP-01-092420 | L33791-1 | N-EtFOSE |
| RC23-SNAIL-COMP-01-092420 | L33791-2 | PFHxS |
| RC23-SNAIL-COMP-01-092420 | L33791-2 | N-EtFOSE |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | PFNA |
| EP27B-SNAIL-ONE-01-092420 | L33791-3 | PFUnA |
| RC4A-AMPH-COMP-01-091820 | L33792-1 | MeFOSAA |
| RC5-AMPH-ONE-01-091820 | L33792-2 | N-EtFOSE |
| RC5-AMPH-TWO-01-092220 | L33792-3 | N-EtFOSE |
| RC6A-AMPH-01-092020 | L33792-4 | PFTrDA |
| RC6A-AMPH-01-092020 | L33792-4 | N-EtFOSE |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | N-EtFOSA |
| RC3A-AMPH-COMP-ONE-01-091920 | L33792-5 | N-EtFOSE |
| RC13-AMPH-01-092120 | L33792-7 | N-EtFOSE |
| RC16A-AMPH-01-091920 | L33792-8 | PFTeDA |
| RC17A-AMPH-01-092120 | L33792-9 | PFUnA |
| EP19-AMPH-01-091920 | L33792-11 | EtFOSAA |
| WL2-AMPH-01-092220 | L33792-12 | N-EtFOSE |
| WL8A-AMPH-01-092520 | L33792-13 | EtFOSAA |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG75489 Report date (mm/dd/yyyy): 2/11/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2021

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. Two results were rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Two results were rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2021

## Data Validation Qualifications for DPWG75489

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18A-FOAM-OLD-01-050520 | L33140-1 | 13C2-PFTeDA | 47.5 | J+ |
| RC18A-FOAM-OLD-01-050520 | L33140-1 | D3-N-MeFOSA | 31.7 | J+ |
| RC18A-FOAM-OLD-01-050520 | L33140-1 | D5-N-EtFOSA | 25.2 | J+ |
| RC18A-FOAM-OLD-01-050520 | L33140-1 | d7-NMe-FOSE | 18.1 | R |
| RC18A-FOAM-OLD-01-050520 | L33140-1 | d9-NEt-FOSE | 19.4 | R |

**SGS AXYS DPWG76270** 5/10/21 **LS**

Initial Cal 2/22/21 **PFBA**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (13C4-PFBA) | PFBA | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | |
| | | | | | |
| 265335 | 11214 | 1.6 | 1.0566 | 1.06 | |
| 285657 | 23973 | 3.2 | 1.0490 | 1.05 | |
| 247666 | 51804 | 8 | 1.0458 | 1.05 | |
| 245774 | 122758 | 20 | 0.9990 | 1.00 | |
| 243172 | 244641 | 40 | 1.0060 | 1.01 | |
| 232710 | 458323 | 80 | 0.9848 | 0.98 | |
| 226018 | 1119980 | 200 | 0.9911 | 0.99 | |
| 172655 | 4123011 | 1000 | 0.9552 | 0.96 | |
| 108520 | 10150232 | 4000 | 0.9353 | 0.94 | |
| | | Avg: | 1.0025 | 1.00 | IS Conc = 40 |
| | | %RSD: | 4.2008 | 4.20 | |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW17A-GW-146-150-01-020421 | PFBA | 6156836 | 53563 | 1 | 8669.59 | 8670 | 0.529 | 0.004 | -2 |
| MW17B-GW-94-98-01-021921 | PFBA | 4544518 | 99402 | 1 | 3334.78 | 3340 | 0.547 | 0.004 | -6 |
| LCS (WG75986-102) | PFBA | 105172 | 214575 | 1 | 19.56 | 19.6 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 4/3/21 23:38 | 141930 | 285174 | 19.858 | 19.9 | 20.00 | 99.29 | 99.3% |
| CCV 4/4/21 3:46 | 147769 | 295831 | 19.930 | 19.9 | 20.00 | 99.649 | 99.6% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG75986-101): | PFBA | 0.119 | <1.6 | verified <1.6 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76270 (Analysis WG75986) dated 4/28/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 5/10/2021 |

The samples listed below were collected at the Project 1007 Site on February 4 through 19, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| MW17A-GW-146-150-01-020421 | | 2/4/21 | L34514-2 |
| MW17A-GW-206-210-01-021521 | | 2/15/21 | L34514-3 |
| MW17B-GW-10-17-01-021821 | | 2/18/21 | L34514-4 |
| MW17B-GW-46-50-01-021821 | | 2/18/21 | L34514-5 |
| MW17B-GW-94-98-01-021921 | | 2/19/21 | L34514-6 |
| **Field QC Samples:** | | | |
| 784025154844 | Trip Blank | 2/19/21 | L34514-1 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✗ | Laboratory and field blanks |
| ✓ | Labeled Analogs (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| NA | Field duplicates |
| ✓ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified J-/UJ due to sample receipt temperature. In addition, one PFNA result was qualified as estimated (J) due to a high ion ratio % recovery. Data qualifications were required as listed in Table 1. Results reported below the quantitation limit were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the validator, but are not shown in Table 1.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact and above the temperature requirement of <6 °C. The samples were received at temperatures of 12.8 °C. The reporting limit for nondetect results were qualified as estimated (UJ) and detects were qualified as estimated biased low (J-).

One trip blank was received, and the sample ID was updated to comply with MPCA chain of custody and EQuIS requirements.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation (occasionally the highest point was not used).

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery.

Two CCVs were recalculated for PFBA during the validation. Results reported are verified to be correct.

**Laboratory and Field Blanks**

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in the method blank.

One trip blank was associated with this sample group. The trip blank results were nondetect.

**Labeled Analogs**

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%.

**MS/MSD Results**

An MS/MSD was not requested for this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not included in this SDG.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFBA. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification.

Ion ratios were within 50% to 150% of the initial calibration ratios, with the exception of PFNA for sample MW17B-GW-46-50-01-021821 (L34514-5) which was 151%. This result was qualified as estimated (J), as both low bias (due to receipt temperature) and high bias (due to ion ratios) is present.

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| MW17B-GW-46-50-01-021821 (L34514-5) | PFNA | ng/L | J | ir |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)

UJ Estimated reporting limit

(2): Reason Codes:

| | |
|---|---|
| Ir | Ion ratio |
| sr | Sample receipt |

**Attachment A**

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76275 (WG75969)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 7.9 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW01-SED-0-12-01-021621 | L34516-1 | 13C2-4:2 FTS | 206 | No Qualifier |
| IW01-SED-24-48-01-021621 | L34516-3 | 13C2-4:2 FTS | 206 | No Qualifier |
| IW02-SED-0-12-01-021621 | L34516-4 | 13C2-4:2 FTS | 203 | No Qualifier |
| IW02-SED-0-12-01-021621 | L34516-4 | D5-N-EtFOSA | 42.7 | UJ |
| IW02-SED-12-24-01-021621 | L34516-5 | 13C2-4:2 FTS | 201 | No Qualifier |
| IW02-SED-24-36-01-021621 | L34516-6 | 13C2-4:2 FTS | 208 | No Qualifier |
| IW03-SED-0-12-01-021621 | L34516-8 | D5-N-EtFOSA | 47.6 | UJ |
| IW04-SED-0-12-01-021621 | L34516-12 | 13C2-4:2 FTS | 204 | No Qualifier |
| IW04-SED-0-12-01-021621 | L34516-12 | D5-N-EtFOSA | 45.4 | UJ |
| IW04-SED-12-24-01-021621 | L34516-13 | D5-N-EtFOSA | 42.6 | UJ |
| IW04-SED-36-48-01-021621 | L34516-15 | 13C2-4:2 FTS | 207 | No Qualifier |
| IW05-SED-0-24-01-021621 | L34516-16 | 13C2-4:2 FTS | 233 | No Qualifier |
| IW05-SED-0-24-01-021621 | L34516-16 | 13C2-8:2 FTS | 205 | No Qualifier |
| IW05-SED-0-24-01-021621 | L34516-16 | D3-N-MeFOSA | 30.0 | J+ |
| IW05-SED-0-24-01-021621 | L34516-16 | D5-N-EtFOSA | 22.1 | J+ |
| IW05-SED-0-24-01-021621 | L34516-16 | d7-NMe-FOSE | 37.8 | UJ |
| IW05-SED-0-24-01-021621 | L34516-16 | d9-NEt-FOSE | 32.3 | UJ |
| IW06-SED-0-12-01-021621 | L34516-18 | 13C2-4:2 FTS | 211 | No Qualifier |
| IW06-SED-0-12-01-021621 | L34516-18 | D5-N-EtFOSA | 43.8 | UJ |
| IW06-SED-12-24-01-021621 | L34516-19 | 13C2-4:2 FTS | 209 | No Qualifier |

**Laboratory control sample (Section B (ii)): The** NFDHA LCS recovery was 48.4% and below the acceptable limits of 50% to 150%. Associated results were nondetect and qualified UJ.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW01-SED-0-12-01-021621 | L34516-1 | PFDoA |
| IW01-SED-12-24-01-021621 | L34516-2 | N-EtFOSA |
| IW02-SED-0-12-01-021621 | L34516-4 | EtFOSAA |
| IW03-SED-0-12-01-021621 | L34516-8 | EtFOSAA |
| IW04-SED-36-48-01-021621 | L34516-15 | PFUnA |
| IW04-SED-36-48-01-021621 | L34516-15 | MeFOSAA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76275 Report date (mm/dd/yyyy): 4/21/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/14/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 7.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☐ | ☒ | No QC samples submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample reciept temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | NFDHA LCS 48.4%<br>See data qualifications summary for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/14/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76277 (WG75971)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 11.9 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV<br>4/6/21 16:34<br>Closing CCV | NFDHA | 64.6% | 70-130% | Associated results were nondetects and were qualified UJ. All results were associated with this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW10-SED-12-24-01-021621 | L34516-41 | 13C2-4:2 FTS | 237 | No Qualifier |
| IW10-SED-12-24-01-021621 | L34516-41 | D5-N-EtFOSA | 46.0 | UJ |
| IW10-SED-12-24-02-021621 | L34516-42 | 13C2-4:2 FTS | 245 | No Qualifier |
| IW10-SED-12-24-02-021621 | L34516-42 | D5-N-EtFOSA | 42.5 | UJ |
| IW10-SED-24-36-01-021621 | L34516-43 | 13C2-4:2 FTS | 219 | No Qualifier |
| IW10-SED-24-36-01-021621 | L34516-43 | D5-N-EtFOSA | 46.7 | UJ |
| IW10-SED-24-36-02-021621 | L34516-44 | 13C2-4:2 FTS | 212 | No Qualifier |
| IW10-SED-24-36-02-021621 | L34516-44 | D5-N-EtFOSA | 46.2 | UJ |
| IW10-SED-36-48-01-021621 | L34516-45 | 13C2-4:2 FTS | 217 | No Qualifier |
| IW10-SED-36-48-02-021621 | L34516-46 | 13C2-4:2 FTS | 206 | No Qualifier |
| IW10-SED-36-48-02-021621 | L34516-46 | D5-N-EtFOSA | 49.9 | UJ |
| IW11-SED-0-12-01-021621 | L34516-47 | 13C2-4:2 FTS | 218 | No Qualifier |
| IW11-SED-0-12-01-021621 | L34516-47 | D3-N-MeFOSA | 46.0 | UJ |
| IW11-SED-0-12-01-021621 | L34516-47 | D5-N-EtFOSA | 32.6 | UJ |
| IW11-SED-12-24-01-021621 | L34516-48 | 13C2-4:2 FTS | 212 | No Qualifier |
| IW11-SED-12-24-01-021621 | L34516-48 | D5-N-EtFOSA | 48.3 | UJ |
| IW11-SED-24-36-01-021621 | L34516-49 | 13C2-4:2 FTS | 209 | No Qualifier |
| IW11-SED-24-36-01-021621 | L34516-49 | D5-N-EtFOSA | 49.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW11-SED-36-48-01-021621 | L34516-50 | 13C2-4:2 FTS | 215 | No Qualifier |
| IW11-SED-36-48-01-021621 | L34516-50 | D5-N-EtFOSA | 37.7 | UJ |
| IW12-SED-0-12-01-021621 | L34516-51 | 13C2-4:2 FTS | 251 | No Qualifier |
| IW12-SED-0-12-01-021621 | L34516-51 | 13C2-8:2 FTS | 201 | No Qualifier |
| IW12-SED-0-12-01-021621 | L34516-51 | D3-N-MeFOSA | 42.8 | UJ |
| IW12-SED-0-12-01-021621 | L34516-51 | D5-N-EtFOSA | 30.0 | J+ |
| IW12-SED-0-12-01-021621 | L34516-51 | d9-NEt-FOSE | 46.6 | UJ |
| IW12-SED-12-24-01-021621 | L34516-52 | 13C2-4:2 FTS | 209 | No Qualifier |
| IW12-SED-12-24-01-021621 | L34516-52 | D5-N-EtFOSA | 34.7 | UJ |
| IW12-SED-24-36-01-021621 | L34516-53 | 13C2-4:2 FTS | 230 | No Qualifier |
| IW12-SED-24-36-01-021621 | L34516-53 | D5-N-EtFOSA | 45.9 | UJ |
| IW12-SED-36-48-01-021621 | L34516-54 | 13C2-4:2 FTS | 225 | No Qualifier |
| IW12-SED-36-48-01-021621 | L34516-54 | D3-N-MeFOSA | 48.1 | UJ |
| IW12-SED-36-48-01-021621 | L34516-54 | D5-N-EtFOSA | 42.6 | UJ |
| IW13-SED-0-12-01-021621 | L34516-55 | 13C2-4:2 FTS | 207 | No Qualifier |
| IW13-SED-0-12-01-021621 | L34516-55 | D5-N-EtFOSA | 38.1 | UJ |
| IW13-SED-12-24-01-021621 | L34516-56 | 13C2-4:2 FTS | 217 | No Qualifier |
| IW13-SED-12-24-01-021621 | L34516-56 | D3-N-MeFOSA | 47.7 | UJ |
| IW13-SED-12-24-01-021621 | L34516-56 | D5-N-EtFOSA | 35.5 | UJ |
| IW13-SED-24-36-01-021621 | L34516-57 | 13C2-4:2 FTS | 212 | No Qualifier |
| IW13-SED-24-36-01-021621 | L34516-57 | D5-N-EtFOSA | 46.3 | UJ |
| IW13-SED-36-48-01-021621 | L34516-58 | 13C2-4:2 FTS | 211 | No Qualifier |
| IW14-SED-0-12-01-021621 | L34516-59 | 13C2-4:2 FTS | 223 | No Qualifier |
| IW14-SED-0-12-01-021621 | L34516-59 | D3-N-MeFOSA | 48.1 | J+ |
| IW14-SED-0-12-01-021621 | L34516-59 | D5-N-EtFOSA | 33.2 | J+ |
| IW14-SED-12-24-01-021621 | L34516-60 | 13C2-4:2 FTS | 215 | No Qualifier |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **IW10-SED-24-36-01-021621/IW10-SED-24-36-02-021621:** | | | |
| PFDA | 1.12 | 0.65 | 53.11 |
| PFOS | 170 | 89.1 | 62.4 |
| PFOSA | 1.1 | 0.408 | 91.8 |
| **IW10-SED-36-48-01-021621/IW10-SED-36-48-02-021621:** | | | |
| 6:2 FTS | 0.26 | 1.01 | 118 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW10-SED-12-24-01-021621 | L34516-41 | N-MeFOSA |
| IW10-SED-24-36-01-021621 | L34516-43 | N-MeFOSA |
| IW12-SED-0-12-01-021621 | L34516-51 | MeFOSAA |
| IW13-SED-36-48-01-021621 | L34516-58 | EtFOSAA |
| IW14-SED-0-12-01-021621 | L34516-59 | PFTeDA |
| IW14-SED-12-24-01-021621 | L34516-60 | N-EtFOSA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76277 Report date (mm/dd/yyyy): 4/28/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/14/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 11.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☐ | ☒ | No QC samples (MS/MSDs) submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD<br>FDs:<br>IW10-SED-12-24-02-021621 (L34516-42)<br>IW10-SED-24-36-02-021621 (L34516-44)<br>IW10-SED-36-48-02-021621 (L34516-46) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/14/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76281 (WG75970)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 7.9 °C and 11.9 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Method Blank detections (Section 3Bii):** Detects within 10 times the method blank concentration were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG75970-101 | PFBA | 0.17 J | IW06-SED-36-48-01-021621 (L34516-21)<br>IW07-SED-0-12-01-021621 (L34516-22)<br>IW07-SED-12-24-01-021621 (L34516-23)<br>IW08-SED-12-24-01-021621 (L34516-28)<br>IW08-SED-12-24-02-021621 (L34516-29)<br>IW08-SED-36-48-01-021621 (L34516-32)<br>IW08-SED-36-48-02-021621 (L34516-33)<br>IW08-SED-36-48-03-021621 (L34516-34 (A))<br>IW09-SED-0-12-01-021621 (L34516-35)<br>IW09-SED-0-12-02-021621 (L34516-36)<br>IW09-SED-12-24-01-021621 (L34516-37)<br>IW09-SED-24-36-01-021621 (L34516-38)<br>IW09-SED-36-48-01-021621 (L34516-39) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Detects associated high recoveries were qualified estimated biased low (J-), and nondetects were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW07-SED-0-12-01-021621 | L34516-22 | 13C2-4:2 FTS | 243 | No Qualifier |
| IW07-SED-0-12-01-021621 | L34516-22 | 13C2-6:2 FTS | 322 | No Qualifier |
| IW07-SED-0-12-01-021621 | L34516-22 | D3-N-MeFOSA | 40.2 | J+ |
| IW07-SED-0-12-01-021621 | L34516-22 | D5-N-EtFOSA | 27.8 | J+ |
| IW07-SED-0-12-01-021621 | L34516-22 | d9-NEt-FOSE | 41.2 | UJ |
| IW07-SED-12-24-01-021621 | L34516-23 | D3-N-MeFOSA | 45.2 | J+ |
| IW07-SED-12-24-01-021621 | L34516-23 | D5-N-EtFOSA | 34.3 | J+ |
| IW07-SED-36-48-01-021621 | L34516-25 | D5-N-EtFOSA | 48.7 | UJ |
| IW08-SED-0-12-01-021621 | L34516-26 | 13C2-4:2 FTS | 232 | No Qualifier |
| IW08-SED-0-12-01-021621 | L34516-26 | D3-N-MeFOSA | 41.8 | J+ |
| IW08-SED-0-12-01-021621 | L34516-26 | D5-N-EtFOSA | 29.1 | J+ |
| IW08-SED-0-12-02-021621 | L34516-27 | 13C2-4:2 FTS | 208 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW08-SED-0-12-02-021621 | L34516-27 | D3-N-MeFOSA | 47.4 | J+ |
| IW08-SED-0-12-02-021621 | L34516-27 | D5-N-EtFOSA | 37.2 | UJ |
| IW08-SED-12-24-01-021621 | L34516-28 | 13C2-4:2 FTS | 215 | No Qualifier |
| IW08-SED-36-48-03-021621 | L34516-34 (A) | 13C2-4:2 FTS | 220 | No Qualifier |
| IW09-SED-0-12-01-021621 | L34516-35 | 13C2-4:2 FTS | 214 | No Qualifier |
| IW09-SED-0-12-01-021621 | L34516-35 | D5-N-EtFOSA | 43.3 | J+ |
| IW09-SED-0-12-02-021621 | L34516-36 | D5-N-EtFOSA | 49.5 | J+ |
| IW09-SED-36-48-01-021621 | L34516-39 | 13C2-4:2 FTS | 201 | No Qualifier |
| IW10-SED-0-12-01-021621 | L34516-40 | 13C2-4:2 FTS | 312 | No Qualifier |
| IW10-SED-0-12-01-021621 | L34516-40 | 13C2-6:2 FTS | 217 | No Qualifier |
| IW10-SED-0-12-01-021621 | L34516-40 | 13C2-8:2 FTS | 202 | No Qualifier |
| IW10-SED-0-12-01-021621 | L34516-40 | D5-N-EtFOSA | 38.4 | UJ |
| IW10-SED-0-12-01-021621 | L34516-40 | D5-EtFOSAA | 238 | J- |

**Matrix spike Matrix Spike Duplicate (Section 6Av and Bi):** The matrix spike recovery and RPD were outside acceptable limits as summarized in the table below. The NFDHA results for samples IW08-SED-36-48-01-021621, IW08-SED-36-48-02-021621, and IW08-SED-36-48-03-021621 were nondetect and qualified UJ.

| Analyte | MS/MSD % Recovery | Recovery Limits | RPD (%) | RPD Limit |
|---|---|---|---|---|
| **IW08-SED-36-48-03-021621:** | | | | |
| NFDHA | **44.0/**80.0 | 50 - 150 | **58.4** | 50 |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

The % Moisture results were considerably different for the field duplicate and parent sample identified in the table below. Since the % moisture results are used in calculating the PFAS results on a dry weight basis, this variability is also evident in the PFAS results. Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **IW08-SED-12-24-01-021621/IW08-SED-12-24-02-021621:** | | | |
| PFOS | 13.9 | 24.4 | 54.8 |
| PFDS | 0.186 | 0.432 | 79.6 |
| PFDoS | 0.089 | 0.27 | 100.8 |
| PFOSA | 0.996 | 2.01 | 67.5 |
| EtFOSAA | 0.403 | 0.744 | 59.5 |
| % Moisture | 9.6 | 16.6 | 53.4 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW08-SED-0-12-01-021621 | L34516-26 | N-EtFOSA |
| IW08-SED-0-12-02-021621 | L34516-27 | MeFOSAA |
| IW08-SED-24-36-02-021621 | L34516-31 | PFDoA |
| IW08-SED-36-48-03-021621 | L34516-34 (A) | MeFOSAA |
| IW09-SED-0-12-01-021621 | L34516-35 | N-EtFOSA |
| IW09-SED-12-24-01-021621 | L34516-37 | N-EtFOSA |
| IW09-SED-12-24-01-021621 | L34516-37 | MeFOSAA |
| IW09-SED-24-36-01-021621 | L34516-38 | MeFOSAA |
| IW10-SED-0-12-01-021621 | L34516-40 | MeFOSAA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76281_2 Report date (mm/dd/yyyy): 4/26/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/14/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at elevated temperatures of 7.9 °C and 11.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFBA 0.17 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW08-SED-36-48-03-021621 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | NFDHA MS 44.0% recovery; RPD 58.4%<br>See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD See Section 6Av above.<br>FDs:<br>IW08-SED-0-12-02-021621 (L34516-27)<br>IW08-SED-12-24-02-021621 (L34516-29)<br>IW08-SED-24-36-02-021621 (L34516-31)<br>IW08-SED-36-48-02-021621 (L34516-33)/IW08-SED-36-48-03-021621 (L34516-34 (A)) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | IW09-SED-0-12-02-021621 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | A reduced sample volume was used for sample IW10-SED-0-12-01-021621 due to high analyte concentrations.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/14/2021

**SGS AXYS DPWG76289** 5/23/21 **LS**

Initial Cal 2/22/21 **PFBA**

| Area Counts | | | | |
|---|---|---|---|---|
| IS (13C4-PFBA) | PFBA | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
| | | | | |
| 265335 | 11214 | 1.6 | 1.0566 | 1.06 |
| 285657 | 23973 | 3.2 | 1.0490 | 1.05 |
| 247666 | 51804 | 8 | 1.0458 | 1.05 |
| 245774 | 122758 | 20 | 0.9990 | 1.00 |
| 243172 | 244641 | 40 | 1.0060 | 1.01 |
| 232710 | 458323 | 80 | 0.9848 | 0.98 |
| 226018 | 1119980 | 200 | 0.9911 | 0.99 |
| 172655 | 4123011 | 1000 | 0.9552 | 0.96 |
| 108520 | 10150232 | 4000 | 0.9353 | 0.94 |
| | | Avg: | 1.0025 | 1.00 |
| | | %RSD: | 4.2008 | 4.20 |

IS Conc = 40

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/g | Reported ng/g | Intial volume (L) | Final Volume (L) | Lab ID | % Moist |
|---|---|---|---|---|---|---|---|---|---|---|
| MW17B-SOIL-COMP-10-17-01-021821 | PFBA | 1073 | 171607 | 1 | 0.0553 | <0.147 | 5.440 | 0.004 | -1 | 0.829 |
| LCS (WG75972-102) | PFBA | 87302 | 174423 | 1 | 19.97 | 20 | 1.00 | 0.004 | | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 4/10/21 00:39 | 148523 | 300767 | 19.703 | 19.7 | 20.00 | 98.51 | 98.5% |
| CCV 4/10/21 06:17 | 151378 | 305597 | 19.764 | 19.8 | 20.00 | 98.820 | 98.8% |

| | | Raw Data (ng/g) | Reported (ng/g) | |
|---|---|---|---|---|
| Method Blank ( WG75972-101): | PFBA | 0.038 | <0.16 | verified <0.16 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76289 (Analysis WG75972) dated 4/21/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 5/16/2021 |

The samples listed below were collected at the Project 1007 Site on February 18, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| MW17B-SOIL-COMP-10-17-01-021821 | | 2/18/21 | L34515-1 |
| MW17B-SOIL-COMP-6-10-01-021821 | | 2/18/21 | L34515-2 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and field blanks |
| ✗ | Labeled Analogs (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| NA | Field duplicates |
| ✓ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified UJ due to sample receipt temperature. In addition, one EtFOSA result was qualified as estimated (UJ) due to a low labeled analog % recovery. Data qualifications were required as listed in Table 1.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact and above the temperature requirement of <6 °C. The samples were received at temperatures of 12.7 °C. The results for samples reported in this SDG were nondetect and the reporting limits were qualified as estimated (UJ).

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

### Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation (occasionally the highest point was not used).

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries for target compounds was not provided.

Two CCVs were recalculated for PFBA during the validation. Results reported are verified to be correct.

### Laboratory and Field Blanks

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in the method blank.

One trip blank was associated with this sample group per CoC (L34514-1). The trip blank results were nondetect.

### Labeled Analogs

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, except as listed in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW17B-SOIL-COMP-10-17-01-021821 | L34515-1 | 13C2-4:2 FTS | 227 | No Qualifier |
| MW17B-SOIL-COMP-10-17-01-021821 | L34515-1 | D5-N-EtFOSA | 45.8 | UJ |

**MS/MSD Results**

An MS/MSD was not requested for this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not included in this SDG.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFBA. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | All PFAS Analytes | ng/g | UJ | sr |
| MW17B-SOIL-COMP-10-17-01-021821 (L34515-1) | EtFOSA | ng/g | UJ | s |

(1): Data Validation Qualifiers:

UJ Estimated reporting limit

(2): Reason Codes:

sr Sample receipt

s Surrogate (labeled analog)

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76302 (WG75972)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 8.4 °C and 11.9 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW14-SED-24-36-01-021621 | L34516-61 | D5-N-EtFOSA | 49.3 | J+ |
| IW14-SED-36-48-01-021621 | L34516-62 | 13C2-4:2 FTS | 206 | No Qualifier |
| IW15-SED-0-12-01-021621 | L34516-63 | 13C2-4:2 FTS | 235 | No Qualifier |
| IW15-SED-0-12-01-021621 | L34516-63 | D3-N-MeFOSA | 36.1 | J+ |
| IW15-SED-0-12-01-021621 | L34516-63 | D5-N-EtFOSA | 26.7 | J+ |
| IW15-SED-12-24-01-021621 | L34516-64 | D5-N-EtFOSA | 44.8 | UJ |
| IW15-SED-24-36-01-021621 | L34516-65 | 13C2-4:2 FTS | 209 | No Qualifier |
| IW15-SED-24-36-01-021621 | L34516-65 | D5-N-EtFOSA | 45.9 | J+ |
| IW15-SED-36-48-01-021621 | L34516-66 | D3-N-MeFOSA | 46.8 | J+ |
| IW15-SED-36-48-01-021621 | L34516-66 | D5-N-EtFOSA | 36.5 | J+ |
| IW16-SED-0-12-01-021621 | L34516-67 | 13C2-4:2 FTS | 265 | No Qualifier |
| IW16-SED-0-12-01-021621 | L34516-67 | D5-N-EtFOSA | 39.6 | UJ |
| IW16-SED-12-24-01-021621 | L34516-68 | 13C2-4:2 FTS | 235 | No Qualifier |
| IW16-SED-24-48-01-021621 | L34516-69 | 13C2-4:2 FTS | 213 | No Qualifier |
| IW16-SED-24-48-01-021621 | L34516-69 | D5-N-EtFOSA | 49.2 | UJ |
| IW17-SED-0-12-01-021721 | L34516-70 | D5-N-EtFOSA | 40.5 | UJ |
| IW17-SED-24-36-01-021721 | L34516-72 | D5-N-EtFOSA | 47.4 | UJ |
| IW17-SED-36-48-01-021721 | L34516-73 | 13C2-4:2 FTS | 209 | No Qualifier |
| IW17-SED-36-48-01-021721 | L34516-73 | D5-N-EtFOSA | 49.3 | UJ |
| IW18-SED-0-12-01-021721 | L34516-74 | D5-N-EtFOSA | 44.9 | UJ |
| IW18-SED-12-24-01-021721 | L34516-75 | 13C2-4:2 FTS | 209 | No Qualifier |
| IW18-SED-36-48-01-021721 | L34516-77 | 13C2-4:2 FTS | 205 | No Qualifier |
| IW19-SED-0-12-01-021621 | L34516-78 | 13C2-PFTeDA | 47.2 | J+<br>PFTrDA: J+ |
| IW19-SED-0-12-01-021621 | L34516-78 | 13C2-4:2 FTS | 216 | No Qualifier |
| IW19-SED-0-12-01-021621 | L34516-78 | D5-N-EtFOSA | 36.3 | UJ |
| IW19-SED-0-12-01-021621 | L34516-78 | D3-N-MeFOSA | 25.5 | UJ |
| IW19-SED-0-12-01-021621 | L34516-78 | d7-NMe-FOSE | 45.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW19-SED-0-12-01-021621 | L34516-78 | d9-NEt-FOSE | 42.4 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW14-SED-24-36-01-021621 | L34516-61 | PFDA |
| IW15-SED-36-48-01-021621 | L34516-66 | N-EtFOSA |
| IW16-SED-0-12-01-021621 | L34516-67 | PFTrDA |
| IW16-SED-12-24-01-021621 | L34516-68 | N-MeFOSAA |
| IW17-SED-0-12-01-021721 | L34516-70 | PFDoA |
| IW17-SED-0-12-01-021721 | L34516-70 | PFUnA |
| IW17-SED-36-48-01-021721 | L34516-73 | PFHxA |
| IW19-SED-0-12-01-021621 | L34516-78 | PFTrDA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76302 Report date (mm/dd/yyyy): 4/21/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/18/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at elevated temperatures of 8.4 °C and 11.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | A QC sample (MS/MSD) was not submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/18/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76378 (WG75993)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 8.4 °C and 7.7 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 4/14/21 16:56 Opening CCV | NFDHA | 53.6% | 70-130% | Associated results were nondetects and were qualified UJ. All results were associated with this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Detects associated high recoveries were qualified estimated biased low (J-), and nondetects were acceptable without qualification. One result was rejected due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW24-SED-0-12-01-021721 | L34519-1 | 13C2-4:2 FTS | 215 | No Qualifier |
| IW24-SED-0-12-01-021721 | L34519-1 | D3-N-MeFOSA | 47.8 | J+ |
| IW24-SED-0-12-01-021721 | L34519-1 | D5-N-EtFOSA | 34.7 | J+ |
| IW24-SED-12-24-01-021721 | L34519-2 | 13C2-4:2 FTS | 221 | No Qualifier |
| IW24-SED-24-36-01-021721 | L34519-3 | 13C2-4:2 FTS | 237 | No Qualifier |
| IW24-SED-36-48-01-021721 | L34519-4 | 13C2-4:2 FTS | 223 | No Qualifier |
| IW25-SED-0-12-01-021721 | L34519-5 | 13C2-4:2 FTS | 214 | No Qualifier |
| IW25-SED-0-12-01-021721 | L34519-5 | D3-N-MeFOSA | 39.6 | J+ |
| IW25-SED-0-12-01-021721 | L34519-5 | D5-N-EtFOSA | 28.9 | J+ |
| IW25-SED-12-24-01-021721 | L34519-6 | 13C2-4:2 FTS | 216 | No Qualifier |
| IW25-SED-12-24-01-021721 | L34519-6 | D5-N-EtFOSA | 46.8 | J+ |
| IW25-SED-24-36-01-021721 | L34519-7 | 13C2-4:2 FTS | 204 | No Qualifier |
| IW25-SED-24-36-01-021721 | L34519-7 | D5-N-EtFOSA | 43.5 | J+ |
| IW25-SED-36-48-01-021721 | L34519-8 | 13C2-4:2 FTS | 204 | No Qualifier |
| IW26-SED-0-12-01-021721 | L34519-9 | 13C2-4:2 FTS | 258 | No Qualifier |
| IW26-SED-0-12-01-021721 | L34519-9 | 13C2-6:2 FTS | 212 | No Qualifier |
| IW26-SED-0-12-01-021721 | L34519-9 | D3-N-MeFOSA | 35.9 | UJ |
| IW26-SED-0-12-01-021721 | L34519-9 | D5-N-EtFOSA | 23.7 | UJ |
| IW26-SED-0-12-01-021721 | L34519-9 | d9-NEt-FOSE | 47.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW26-SED-12-24-01-021721 | L34519-10 | 13C2-4:2 FTS | 202 | No Qualifier |
| IW26-SED-12-24-01-021721 | L34519-10 | D5-N-EtFOSA | 41.4 | UJ |
| IW26-SED-24-36-01-021721 | L34519-13 | D5-N-EtFOSA | 44.5 | UJ |
| IW26-SED-36-48-01-021721 | L34519-14 | 13C2-4:2 FTS | 217 | No Qualifier |
| IW27-SED-0-12-01-021721 | L34519-15 | 13C2-4:2 FTS | 236 | No Qualifier |
| IW27-SED-0-12-01-021721 | L34519-15 | D3-N-MeFOSA | 25.5 | J+ |
| IW27-SED-0-12-01-021721 | L34519-15 | D5-N-EtFOSA | 19.9 | R |
| IW27-SED-0-12-01-021721 | L34519-15 | D5-EtFOSAA | 205 | J- |
| IW27-SED-0-12-01-021721 | L34519-15 | d7-NMe-FOSE | 40.6 | UJ |
| IW27-SED-0-12-01-021721 | L34519-15 | d9-NEt-FOSE | 36.5 | UJ |
| IW27-SED-12-24-01-021721 | L34519-16 | D3-N-MeFOSA | 49.3 | J+ |
| IW27-SED-12-24-01-021721 | L34519-16 | D5-N-EtFOSA | 37.8 | J+ |
| IW27-SED-24-36-01-021721 | L34519-17 | 13C2-4:2 FTS | 213 | No Qualifier |
| IW27-SED-24-36-01-021721 | L34519-17 | D5-N-EtFOSA | 45.5 | UJ |
| IW27-SED-36-48-01-021721 | L34519-18 | 13C2-4:2 FTS | 202 | No Qualifier |
| IW27-SED-36-48-01-021721 | L34519-18 | D3-N-MeFOSA | 48.9 | J+ |
| IW27-SED-36-48-01-021721 | L34519-18 | D5-N-EtFOSA | 38.6 | UJ |
| IW27-SED-24-36-03-021721 | L34519-19 (A) | 13C2-4:2 FTS | 218 | No Qualifier |
| IW28-SED-0-12-01-021721 | L34519-20 | 13C2-4:2 FTS | 265 | No Qualifier |
| IW28-SED-0-12-01-021721 | L34519-20 | 13C2-6:2 FTS | 206 | No Qualifier |
| IW28-SED-0-12-01-021721 | L34519-20 | D3-N-MeFOSA | 36.3 | J+ |
| IW28-SED-0-12-01-021721 | L34519-20 | D5-N-EtFOSA | 25.7 | J+ |
| IW28-SED-0-12-01-021721 | L34519-20 | D5-EtFOSAA | 208 | J- |
| IW28-SED-0-12-01-021721 | L34519-20 | d9-NEt-FOSE | 49.4 | UJ |
| IW28-SED-12-24-01-021721 | L34519-21 | 13C2-4:2 FTS | 217 | No Qualifier |
| IW28-SED-12-24-01-021721 | L34519-21 | D5-N-EtFOSA | 41.6 | J+ |

**Matrix spike Matrix Spike Duplicate (Section 6Av):** The MS/MSD recoveries were outside acceptable limits as summarized in the table below. Results qualified are discussed in the table below.

| Analyte | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **IW27-SED-24-36-03-021721:** | | | |
| PFOA | 58.7/**42.1** | 50 - 150 | Associated results (IW27-SED-24-36-01-021721 and IW27-SED-24-36-03-021721) were qualified as estimated biased low (J-). |
| PFOS | **3.02/158** | 50 - 150 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **IW27-SED-24-36-01-021721/IW27-SED-24-36-03-021721:** | | | |
| PFUnA | 0.209 | 0.125 | 50.30 |
| PFNS | 0.411 | 0.224 | 58.90 |
| PFDS | 0.316 | 0.181 | 54.33 |
| MeFOSAA | 0.202 | 0.096 | 71.14 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentrations and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW25-SED-24-36-01-021721 | L34519-7 | MeFOSAA |
| IW28-SED-12-24-01-021721 | L34519-21 | MeFOSAA |
| IW28-SED-24-36-01-021721 | L34519-22 | N-EtFOSA |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76378 Report date (mm/dd/yyyy): 4/27/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/17/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at elevated temperatures of 8.4 °C and 7.7 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | Samples L34519-11 and L34519-12 were not received. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. One result was rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW27-SED-24-36-03-021721 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>FDs:<br>IW27-SED-24-36-03-021721 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Reduced sample volume used for sample IW28-SED-0-12-01-021721 due to high analyte concentrations.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**One result was rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/17/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76405 (WG75973)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 8.4 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Method blank detections (Section 3Bii):** Detects within 10 times the method blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| WG75973-101 | 6:2 FTS | 0.421 J | IW21-SED-12-24-01-021621 (L34516-88) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification. One result was rejected due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW19-SED-12-24-01-021621 | L34516-79 | D5-N-EtFOSA | 45.6 | UJ |
| IW19-SED-24-36-01-021621 | L34516-80 | D5-N-EtFOSA | 47.9 | UJ |
| IW19-SED-36-48-01-021621 | L34516-81 | D3-N-MeFOSA | 49.0 | J+ |
| IW19-SED-36-48-01-021621 | L34516-81 | D5-N-EtFOSA | 38.7 | UJ |
| IW19-SED-12-24-03-021621 | L34516-82 (A) | D3-N-MeFOSA | 47.7 | J+ |
| IW19-SED-12-24-03-021621 | L34516-82 (A) | D5-N-EtFOSA | 36.5 | UJ |
| IW20-SED-0-12-01-021621 | L34516-83 | 13C2-4:2 FTS | 247 | No Qualifier |
| IW20-SED-0-12-01-021621 | L34516-83 | D3-N-MeFOSA | 31.6 | UJ |
| IW20-SED-0-12-01-021621 | L34516-83 | D5-N-EtFOSA | 19.4 | R |
| IW20-SED-0-12-01-021621 | L34516-83 | d7-NMe-FOSE | 38.7 | UJ |
| IW20-SED-0-12-01-021621 | L34516-83 | d9-NEt-FOSE | 34.6 | UJ |
| IW20-SED-12-24-01-021621 | L34516-84 | 13C2-4:2 FTS | 204 | No Qualifier |
| IW20-SED-12-24-01-021621 | L34516-84 | D5-N-EtFOSA | 40.2 | UJ |
| IW20-SED-24-36-01-021621 | L34516-85 | 13C2-4:2 FTS | 208 | No Qualifier |
| IW20-SED-24-36-01-021621 | L34516-85 | D5-N-EtFOSA | 44.6 | UJ |
| IW20-SED-36-48-01-021621 | L34516-86 | 13C2-4:2 FTS | 203 | No Qualifier |
| IW20-SED-36-48-01-021621 | L34516-86 | D5-N-EtFOSA | 44.5 | UJ |
| IW21-SED-0-12-01-021621 | L34516-87 | 13C2-4:2 FTS | 267 | No Qualifier |
| IW21-SED-0-12-01-021621 | L34516-87 | D3-N-MeFOSA | 40.7 | J+ |
| IW21-SED-0-12-01-021621 | L34516-87 | D5-N-EtFOSA | 27.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW21-SED-0-12-01-021621 | L34516-87 | d9-NEt-FOSE | 48.9 | UJ |
| IW21-SED-12-24-01-021621 | L34516-88 | 13C2-4:2 FTS | 215 | No Qualifier |
| IW21-SED-12-24-01-021621 | L34516-88 | D5-N-EtFOSA | 41.8 | UJ |
| IW21-SED-24-36-01-021621 | L34516-89 | D5-N-EtFOSA | 41.2 | UJ |
| IW21-SED-36-48-01-021621 | L34516-90 | 13C2-4:2 FTS | 231 | No Qualifier |
| IW21-SED-36-48-01-021621 | L34516-90 | D3-N-MeFOSA | 44.8 | UJ |
| IW21-SED-36-48-01-021621 | L34516-90 | D5-N-EtFOSA | 36.7 | UJ |
| IW22-SED-0-12-01-021621 | L34516-91 | 13C2-4:2 FTS | 211 | No Qualifier |
| IW22-SED-0-12-01-021621 | L34516-91 | D5-N-EtFOSA | 47.8 | UJ |
| IW22-SED-12-24-01-021621 | L34516-92 | 13C2-4:2 FTS | 218 | No Qualifier |
| IW22-SED-12-24-01-021621 | L34516-92 | D5-N-EtFOSA | 48.3 | UJ |
| IW22-SED-24-36-01-021621 | L34516-93 | D3-N-MeFOSA | 48.1 | UJ |
| IW22-SED-24-36-01-021621 | L34516-93 | D5-N-EtFOSA | 38.8 | UJ |
| IW22-SED-36-48-01-021621 | L34516-94 | D5-N-EtFOSA | 44.4 | UJ |
| IW23-SED-24-36-01-021721 | L34516-95 | 13C2-4:2 FTS | 221 | No Qualifier |
| IW23-SED-24-36-01-021721 | L34516-95 | D3-N-MeFOSA | 46.8 | J+ |
| IW23-SED-24-36-01-021721 | L34516-95 | D5-N-EtFOSA | 37.5 | J+ |
| IW23-SED-36-48-01-021721 | L34516-96 | 13C2-4:2 FTS | 215 | No Qualifier |
| IW23-SED-36-48-01-021721 | L34516-96 | D3-N-MeFOSA | 46.3 | J+ |
| IW23-SED-36-48-01-021721 | L34516-96 | D5-N-EtFOSA | 36.3 | J+ |
| IW23-SED-0-24-01-021721 | L34516-97 | 13C2-4:2 FTS | 223 | No Qualifier |
| IW23-SED-0-24-01-021721 | L34516-97 | D5-N-EtFOSA | 39.0 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentrations and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW19-SED-12-24-01-021621 | L34516-79 | N-MeFOSAA |
| IW21-SED-0-12-01-021621 | L34516-87 | PFPeS |
| IW21-SED-12-24-01-021621 | L34516-88 | N-MeFOSAA |
| IW22-SED-36-48-01-021621 | L34516-94 | N-EtFOSAA |
| IW23-SED-24-36-01-021721 | L34516-95 | N-EtFOSA |
| IW23-SED-36-48-01-021721 | L34516-96 | N-EtFOSA |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76405 Report date (mm/dd/yyyy): 4/28/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/18/2021

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 8.4 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 0.421 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. One result was rejected (R) due to a severely low labeled analog recovery. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW19-SED-12-24-03-021621 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>FDs - ok:<br>IW19-SED-12-24-03-021621 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Reduced sample volume used for sample IW21-SED-0-12-01-021621 due to high analyte concentrations.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**One result was rejected (R) due to a severely low labeled analog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/18/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76440 (WG75995)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 7.3 °C and 7.7 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration Date | Analyte | %R | %R Limit | Qualifiers |
|---|---|---|---|---|
| 4/20/21 13:39 | NFDHA | 65.6 | 70-130 | All NFDHA results were nondetect and associated with this CCV. Results were qualified UJ. |
| 4/23/21 18:45 | NFDHA | 69.3 | 70-130 | Only PFOS dilutions reported from 4/23/21 analyses. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Detects associated high recoveries were qualified estimated biased low (J-), and nondetects were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW34-SED-0-12-01-021721 | L34519-43 | 13C2-4:2 FTS | 272 | No Qualifier |
| IW34-SED-0-12-01-021721 | L34519-43 | 13C2-6:2 FTS | 277 | No Qualifier |
| IW34-SED-0-12-01-021721 | L34519-43 | 13C2-8:2 FTS | 211 | No Qualifier |
| IW34-SED-0-12-01-021721 | L34519-43 | D3-N-MeFOSA | 40.5 | J+ |
| IW34-SED-0-12-01-021721 | L34519-43 | D5-N-EtFOSA | 33.3 | J+ |
| IW34-SED-12-24-01-021721 | L34519-44 | 13C2-4:2 FTS | 312 | No Qualifier |
| IW34-SED-12-24-01-021721 | L34519-44 | 13C2-6:2 FTS | 267 | No Qualifier |
| IW34-SED-12-24-01-021721 | L34519-44 | 13C2-8:2 FTS | 238 | No Qualifier |
| IW34-SED-24-36-01-021721 | L34519-45 | 13C2-4:2 FTS | 287 | No Qualifier |
| IW34-SED-24-36-01-021721 | L34519-45 | 13C2-6:2 FTS | 259 | No Qualifier |
| IW34-SED-24-36-01-021721 | L34519-45 | 13C2-8:2 FTS | 227 | No Qualifier |
| IW34-SED-24-36-01-021721 | L34519-45 | D3-N-MeFOSA | 46.2 | J+ |
| IW34-SED-24-36-01-021721 | L34519-45 | D5-N-EtFOSA | 42.2 | J+ |
| IW34-SED-36-48-01-021721 | L34519-46 | 13C2-4:2 FTS | 293 | No Qualifier |
| IW34-SED-36-48-01-021721 | L34519-46 | 13C2-6:2 FTS | 275 | No Qualifier |
| IW34-SED-36-48-01-021721 | L34519-46 | 13C2-8:2 FTS | 243 | No Qualifier |
| IW35-SED-0-12-01-021721 | L34519-47 | 13C2-4:2 FTS | 309 | No Qualifier |
| IW35-SED-0-12-01-021721 | L34519-47 | 13C2-6:2 FTS | 283 | No Qualifier |
| IW35-SED-0-12-01-021721 | L34519-47 | 13C2-8:2 FTS | 264 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW35-SED-0-12-01-021721 | L34519-47 | D3-N-MeFOSA | 27.3 | J+ |
| IW35-SED-0-12-01-021721 | L34519-47 | D5-N-EtFOSA | 20.0 | J+ |
| IW35-SED-0-12-01-021721 | L34519-47 | d7-NMe-FOSE | 36.1 | UJ |
| IW35-SED-0-12-01-021721 | L34519-47 | d9-NEt-FOSE | 35.0 | UJ |
| IW35-SED-12-24-01-021721 | L34519-48 | 13C2-4:2 FTS | 312 | No Qualifier |
| IW35-SED-12-24-01-021721 | L34519-48 | 13C2-6:2 FTS | 267 | No Qualifier |
| IW35-SED-12-24-01-021721 | L34519-48 | 13C2-8:2 FTS | 238 | No Qualifier |
| IW35-SED-12-24-01-021721 | L34519-48 | D5-N-EtFOSA | 42.2 | J+ |
| IW35-SED-24-36-01-021721 | L34519-49 | 13C2-4:2 FTS | 283 | No Qualifier |
| IW35-SED-24-36-01-021721 | L34519-49 | 13C2-6:2 FTS | 267 | No Qualifier |
| IW35-SED-24-36-01-021721 | L34519-49 | 13C2-8:2 FTS | 245 | No Qualifier |
| IW35-SED-24-36-01-021721 | L34519-49 | D5-N-EtFOSA | 47.8 | J+ |
| IW35-SED-36-48-01-021721 | L34519-50 | 13C2-4:2 FTS | 313 | No Qualifier |
| IW35-SED-36-48-01-021721 | L34519-50 | 13C2-6:2 FTS | 264 | No Qualifier |
| IW35-SED-36-48-01-021721 | L34519-50 | 13C2-8:2 FTS | 267 | No Qualifier |
| IW35-SED-36-48-01-021721 | L34519-50 | D5-N-EtFOSA | 47.6 | UJ |
| IW36-SED-0-12-01-021721 | L34519-51 | 13C2-4:2 FTS | 348 | No Qualifier |
| IW36-SED-0-12-01-021721 | L34519-51 | 13C2-6:2 FTS | 303 | No Qualifier |
| IW36-SED-0-12-01-021721 | L34519-51 | 13C2-8:2 FTS | 274 | No Qualifier |
| IW36-SED-0-12-01-021721 | L34519-51 | D3-N-MeFOSA | 48.3 | J+ |
| IW36-SED-0-12-01-021721 | L34519-51 | D5-N-EtFOSA | 32.3 | J+ |
| IW36-SED-0-12-01-021721 | L34519-51 | d9-NEt-FOSE | 45.2 | UJ |
| IW36-SED-12-24-01-021721 | L34519-52 | 13C2-4:2 FTS | 326 | No Qualifier |
| IW36-SED-12-24-01-021721 | L34519-52 | 13C2-6:2 FTS | 296 | No Qualifier |
| IW36-SED-12-24-01-021721 | L34519-52 | 13C2-8:2 FTS | 280 | No Qualifier |
| IW36-SED-12-24-01-021721 | L34519-52 | D5-N-EtFOSA | 45.4 | UJ |
| IW36-SED-24-36-01-021721 | L34519-53 | 13C2-4:2 FTS | 302 | No Qualifier |
| IW36-SED-24-36-01-021721 | L34519-53 | 13C2-6:2 FTS | 279 | No Qualifier |
| IW36-SED-24-36-01-021721 | L34519-53 | 13C2-8:2 FTS | 256 | No Qualifier |
| IW36-SED-24-36-01-021721 | L34519-53 | D5-N-EtFOSA | 48.2 | UJ |
| IW36-SED-36-48-01-021721 | L34519-54 | 13C2-4:2 FTS | 316 | No Qualifier |
| IW36-SED-36-48-01-021721 | L34519-54 | 13C2-6:2 FTS | 281 | No Qualifier |
| IW36-SED-36-48-01-021721 | L34519-54 | 13C2-8:2 FTS | 248 | No Qualifier |
| IW37-SED-0-12-01-021721 | L34519-55 | 13C2-4:2 FTS | 351 | No Qualifier |
| IW37-SED-0-12-01-021721 | L34519-55 | 13C2-6:2 FTS | 286 | No Qualifier |
| IW37-SED-0-12-01-021721 | L34519-55 | 13C2-8:2 FTS | 280 | No Qualifier |
| IW37-SED-0-12-01-021721 | L34519-55 | D3-N-MeFOSA | 43.5 | J+ |
| IW37-SED-0-12-01-021721 | L34519-55 | D5-N-EtFOSA | 32.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW37-SED-12-24-01-021721 | L34519-56 | 13C2-4:2 FTS | 337 | No Qualifier |
| IW37-SED-12-24-01-021721 | L34519-56 | 13C2-6:2 FTS | 315 | No Qualifier |
| IW37-SED-12-24-01-021721 | L34519-56 | 13C2-8:2 FTS | 276 | No Qualifier |
| IW37-SED-24-36-01-021721 | L34519-57 | 13C2-4:2 FTS | 328 | No Qualifier |
| IW37-SED-24-36-01-021721 | L34519-57 | 13C2-6:2 FTS | 274 | No Qualifier |
| IW37-SED-24-36-01-021721 | L34519-57 | 13C2-8:2 FTS | 230 | No Qualifier |
| IW37-SED-36-48-01-021721 | L34519-58 | 13C2-4:2 FTS | 372 | No Qualifier |
| IW37-SED-36-48-01-021721 | L34519-58 | 13C2-6:2 FTS | 307 | No Qualifier |
| IW37-SED-36-48-01-021721 | L34519-58 | 13C2-8:2 FTS | 254 | No Qualifier |
| IW38-SED-0-24-01-021721 | L34519-59 | 13C2-4:2 FTS | 328 | No Qualifier |
| IW38-SED-0-24-01-021721 | L34519-59 | 13C2-6:2 FTS | 311 | J- |
| IW38-SED-0-24-01-021721 | L34519-59 | 13C2-8:2 FTS | 297 | No Qualifier |
| IW38-SED-0-24-01-021721 | L34519-59 | D3-N-MeFOSA | 46.4 | J+ |
| IW38-SED-0-24-01-021721 | L34519-59 | D5-N-EtFOSA | 33.3 | J+ |
| IW38-SED-24-36-01-021721 | L34519-60 | 13C2-4:2 FTS | 381 | No Qualifier |
| IW38-SED-24-36-01-021721 | L34519-60 | 13C2-6:2 FTS | 295 | No Qualifier |
| IW38-SED-24-36-01-021721 | L34519-60 | 13C2-8:2 FTS | 310 | No Qualifier |
| IW38-SED-36-48-01-021721 | L34519-61 | 13C2-4:2 FTS | 352 | No Qualifier |
| IW38-SED-36-48-01-021721 | L34519-61 | 13C2-6:2 FTS | 280 | No Qualifier |
| IW38-SED-36-48-01-021721 | L34519-61 | 13C2-8:2 FTS | 262 | No Qualifier |
| IW39-SED-0-12-01-021721 | L34519-62 | 13C2-4:2 FTS | 307 | No Qualifier |
| IW39-SED-0-12-01-021721 | L34519-62 | 13C2-6:2 FTS | 264 | No Qualifier |
| IW39-SED-0-12-01-021721 | L34519-62 | 13C2-8:2 FTS | 258 | No Qualifier |
| IW39-SED-0-12-01-021721 | L34519-62 | D3-N-MeFOSA | 33.1 | J+ |
| IW39-SED-0-12-01-021721 | L34519-62 | D5-N-EtFOSA | 25.3 | J+ |
| IW39-SED-0-12-01-021721 | L34519-62 | d7-NMe-FOSE | 48.6 | UJ |
| IW39-SED-0-12-01-021721 | L34519-62 | d9-NEt-FOSE | 44.4 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW36-SED-0-12-01-021721 | L34519-51 | N-MeFOSAA |
| IW36-SED-0-12-01-021721 | L34519-51 | PFTeDA |
| IW38-SED-0-24-01-021721 | L34519-59 | N-EtFOSA |
| IW38-SED-0-24-01-021721 | L34519-59 | PFTeDA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76440 Report date (mm/dd/yyyy): 5/5/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/18/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at elevated temperatures of 7.3 °C and 7.7 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | A QC sample (MS/MSD) was not submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | PFOS for sample IW35-SED-0-12-01-021721 was reported from a dilution analysis due to high analyte concentration. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/18/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76483 (WG75997)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 7.3 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ). Detects associated high recoveries were qualified estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW43-SED-36-48-01-021721 | L34519-83 | 13C2-4:2 FTS | 291 | No Qualifier |
| IW43-SED-36-48-01-021721 | L34519-83 | 13C2-6:2 FTS | 260 | J- |
| IW43-SED-36-48-01-021721 | L34519-83 | 13C2-8:2 FTS | 249 | No Qualifier |
| IW43-SED-36-48-01-021721 | L34519-83 | D5-N-EtFOSA | 48.2 | UJ |
| IW43-SED-36-48-02-021721 | L34519-84 | 13C2-4:2 FTS | 299 | No Qualifier |
| IW43-SED-36-48-02-021721 | L34519-84 | 13C2-6:2 FTS | 283 | No Qualifier |
| IW43-SED-36-48-02-021721 | L34519-84 | 13C2-8:2 FTS | 266 | No Qualifier |
| IW43-SED-36-48-02-021721 | L34519-84 | D5-N-EtFOSA | 48.6 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (UJ and J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **IW43-SED-36-48-01-021721/IW43-SED-36-48-02-021721:** | | | |
| 6:2 FTS | 0.555 | 0.137 U | >±RL |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76483 Report date (mm/dd/yyyy): 5/6/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 7.3 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | A QC sample (MS/MSD) was not submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD ok<br>FD: IW43-SED-36-48-02-021721 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76494 (WG75996)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 7.3 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW39-SED-12-24-01-021721 | L34519-63 | 13C2-4:2 FTS | 326 | No Qualifier |
| IW39-SED-12-24-01-021721 | L34519-63 | 13C2-6:2 FTS | 282 | No Qualifier |
| IW39-SED-12-24-01-021721 | L34519-63 | 13C2-8:2 FTS | 234 | No Qualifier |
| IW39-SED-24-36-01-021721 | L34519-64 | 13C2-4:2 FTS | 327 | No Qualifier |
| IW39-SED-24-36-01-021721 | L34519-64 | 13C2-6:2 FTS | 287 | No Qualifier |
| IW39-SED-24-36-01-021721 | L34519-64 | 13C2-8:2 FTS | 243 | No Qualifier |
| IW39-SED-36-48-01-021721 | L34519-65 | 13C2-4:2 FTS | 318 | No Qualifier |
| IW39-SED-36-48-01-021721 | L34519-65 | 13C2-6:2 FTS | 265 | No Qualifier |
| IW39-SED-36-48-01-021721 | L34519-65 | 13C2-8:2 FTS | 229 | No Qualifier |
| IW40-SED-0-12-01-021721 | L34519-66 | 13C2-4:2 FTS | 322 | No Qualifier |
| IW40-SED-0-12-01-021721 | L34519-66 | 13C2-6:2 FTS | 298 | No Qualifier |
| IW40-SED-0-12-01-021721 | L34519-66 | 13C2-8:2 FTS | 251 | No Qualifier |
| IW40-SED-12-24-01-021721 | L34519-67 | 13C2-4:2 FTS | 286 | No Qualifier |
| IW40-SED-12-24-01-021721 | L34519-67 | 13C2-6:2 FTS | 263 | No Qualifier |
| IW40-SED-12-24-01-021721 | L34519-67 | 13C2-8:2 FTS | 245 | No Qualifier |
| IW40-SED-24-48-01-021721 | L34519-68 | 13C2-4:2 FTS | 333 | No Qualifier |
| IW40-SED-24-48-01-021721 | L34519-68 | 13C2-6:2 FTS | 270 | No Qualifier |
| IW40-SED-24-48-01-021721 | L34519-68 | 13C2-8:2 FTS | 258 | No Qualifier |
| IW41-SED-0-12-01-021721 | L34519-69 | 13C2-4:2 FTS | 298 | No Qualifier |
| IW41-SED-0-12-01-021721 | L34519-69 | 13C2-6:2 FTS | 292 | No Qualifier |
| IW41-SED-0-12-01-021721 | L34519-69 | 13C2-8:2 FTS | 244 | No Qualifier |
| IW41-SED-0-12-01-021721 | L34519-69 | D5-N-EtFOSA | 48.7 | UJ |
| IW41-SED-12-24-01-021721 | L34519-70 | 13C2-4:2 FTS | 312 | No Qualifier |
| IW41-SED-12-24-01-021721 | L34519-70 | 13C2-6:2 FTS | 286 | No Qualifier |
| IW41-SED-12-24-01-021721 | L34519-70 | 13C2-8:2 FTS | 247 | No Qualifier |
| IW41-SED-24-48-01-021721 | L34519-71 | 13C2-4:2 FTS | 305 | No Qualifier |
| IW41-SED-24-48-01-021721 | L34519-71 | 13C2-6:2 FTS | 258 | No Qualifier |
| IW41-SED-24-48-01-021721 | L34519-71 | 13C2-8:2 FTS | 257 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW42-SED-0-12-01-021721 | L34519-72 | 13C2-4:2 FTS | 355 | No Qualifier |
| IW42-SED-0-12-01-021721 | L34519-72 | 13C2-6:2 FTS | 304 | No Qualifier |
| IW42-SED-0-12-01-021721 | L34519-72 | 13C2-8:2 FTS | 266 | No Qualifier |
| IW42-SED-0-12-01-021721 | L34519-72 | D5-N-EtFOSA | 49.2 | UJ |
| IW42-SED-12-24-01-021721 | L34519-73 | 13C2-4:2 FTS | 326 | No Qualifier |
| IW42-SED-12-24-01-021721 | L34519-73 | 13C2-6:2 FTS | 287 | No Qualifier |
| IW42-SED-12-24-01-021721 | L34519-73 | 13C2-8:2 FTS | 285 | No Qualifier |
| IW42-SED-24-36-01-021721 | L34519-74 | 13C2-4:2 FTS | 345 | No Qualifier |
| IW42-SED-24-36-01-021721 | L34519-74 | 13C2-6:2 FTS | 298 | No Qualifier |
| IW42-SED-24-36-01-021721 | L34519-74 | 13C2-8:2 FTS | 269 | No Qualifier |
| IW42-SED-24-36-01-021721 | L34519-74 | D5-N-EtFOSA | 49.7 | UJ |
| IW42-SED-36-48-01-021721 | L34519-75 | 13C2-4:2 FTS | 367 | No Qualifier |
| IW42-SED-36-48-01-021721 | L34519-75 | 13C2-6:2 FTS | 292 | No Qualifier |
| IW42-SED-36-48-01-021721 | L34519-75 | 13C2-8:2 FTS | 270 | No Qualifier |
| IW43-SED-0-12-01-021721 | L34519-76 | 13C2-4:2 FTS | 338 | No Qualifier |
| IW43-SED-0-12-01-021721 | L34519-76 | 13C2-6:2 FTS | 298 | No Qualifier |
| IW43-SED-0-12-01-021721 | L34519-76 | 13C2-8:2 FTS | 238 | No Qualifier |
| IW43-SED-0-12-02-021721 | L34519-77 | 13C2-4:2 FTS | 338 | No Qualifier |
| IW43-SED-0-12-02-021721 | L34519-77 | 13C2-6:2 FTS | 322 | No Qualifier |
| IW43-SED-0-12-02-021721 | L34519-77 | 13C2-8:2 FTS | 292 | No Qualifier |
| IW43-SED-0-12-03-021721 | L34519-78 (A) | 13C2-4:2 FTS | 385 | No Qualifier |
| IW43-SED-0-12-03-021721 | L34519-78 (A) | 13C2-6:2 FTS | 329 | No Qualifier |
| IW43-SED-0-12-03-021721 | L34519-78 (A) | 13C2-8:2 FTS | 318 | No Qualifier |
| IW43-SED-0-12-03-021721 | L34519-78 (A) | D5-EtFOSAA | 216 | No Qualifier |
| IW43-SED-12-24-01-021721 | L34519-79 | 13C2-4:2 FTS | 311 | No Qualifier |
| IW43-SED-12-24-01-021721 | L34519-79 | 13C2-6:2 FTS | 279 | No Qualifier |
| IW43-SED-12-24-01-021721 | L34519-79 | 13C2-8:2 FTS | 252 | No Qualifier |
| IW43-SED-12-24-02-021721 | L34519-80 | 13C2-4:2 FTS | 332 | No Qualifier |
| IW43-SED-12-24-02-021721 | L34519-80 | 13C2-6:2 FTS | 280 | No Qualifier |
| IW43-SED-12-24-02-021721 | L34519-80 | 13C2-8:2 FTS | 291 | No Qualifier |
| IW43-SED-24-36-01-021721 | L34519-81 | 13C2-4:2 FTS | 336 | No Qualifier |
| IW43-SED-24-36-01-021721 | L34519-81 | 13C2-6:2 FTS | 296 | No Qualifier |
| IW43-SED-24-36-01-021721 | L34519-81 | 13C2-8:2 FTS | 258 | No Qualifier |
| IW43-SED-24-36-01-021721 | L34519-81 | D5-EtFOSAA | 42.8 | J+ |
| IW43-SED-24-36-02-021721 | L34519-82 | 13C2-4:2 FTS | 370 | No Qualifier |
| IW43-SED-24-36-02-021721 | L34519-82 | 13C2-6:2 FTS | 312 | No Qualifier |
| IW43-SED-24-36-02-021721 | L34519-82 | 13C2-8:2 FTS | 284 | No Qualifier |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Field Triplicate Result (ng/g) | RPD (%) |
|---|---|---|---|---|
| **IW43-SED-0-12-01-021721/IW43-SED-0-12-02-021721/IW43-SED-0-12-03-021721:** | | | | |
| PFDA | 0.337 | 0.345 | 0.195 | 51.3 |
| **IW43-SED-12-24-01-021721/IW43-SED-12-24-02-021721:** | | | | |
| EtFOSAA | 0.193 | 0.095 J | NA | >±RL |
| **IW43-SED-24-36-01-021721/ IW43-SED-24-36-02-021721:** | | | | |
| PFDS | 0.258 | 0.084 J | NA | >±RL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW39-SED-24-36-01-021721 | L34519-64 | N-EtFOSAA |
| IW40-SED-12-24-01-021721 | L34519-67 | N-MeFOSAA |
| IW41-SED-0-12-01-021721 | L34519-69 | N-EtFOSAA |
| IW42-SED-24-36-01-021721 | L34519-74 | N-EtFOSAA |
| IW43-SED-0-12-02-021721 | L34519-77 | PFDoA |
| IW43-SED-12-24-02-021721 | L34519-80 | PFUnA |
| IW43-SED-24-36-01-021721 | L34519-81 | N-MeFOSAA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76494 Report date (mm/dd/yyyy): 5/7/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 7.3 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW43-SED-0-12-03-021721 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD ok<br>FD: IW43-SED-0-12-02-021721/IW43-SED-0-12-03-021721<br>IW43-SED-12-24-02-021721<br>IW43-SED-24-36-02-021721 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76616 (WGV76352)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 7.4 and 14.1 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/11/21 18:44 | NFDHA | 67.2% | 70-130% | Associated results were nondetects were qualified UJ. All NFDHA results were associated with these CCVs. |
| CCV 5/12/21 00:23 | NFDHA | 58.0% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW09-SED-0-12-01-033121 | L34670-1 | D3-N-MeFOSA | 34.4 | UJ |
| FW09-SED-0-12-01-033121 | L34670-1 | D5-N-EtFOSA | 25.5 | UJ |
| FW09-SED-0-12-01-033121 | L34670-1 | d7-NMe-FOSE | 48.5 | UJ |
| FW09-SED-0-12-01-033121 | L34670-1 | d9-NEt-FOSE | 41.2 | UJ |
| FW10-SED-0-12-01-033121 | L34670-2 | 13C2-PFTeDA | 46.6 | UJ |
| FW10-SED-0-12-01-033121 | L34670-2 | 13C2-4:2 FTS | 264 | No Qualifier |
| FW10-SED-0-12-01-033121 | L34670-2 | D3-N-MeFOSA | 23.2 | UJ |
| FW10-SED-0-12-01-033121 | L34670-2 | D5-N-EtFOSA | 16.4 | UJ |
| FW10-SED-0-12-01-033121 | L34670-2 | d7-NMe-FOSE | 30.9 | UJ |
| FW10-SED-0-12-01-033121 | L34670-2 | d9-NEt-FOSE | 25.1 | UJ |
| FW11-SED-0-12-01-033121 | L34670-3 | 13C2-4:2 FTS | 254 | No Qualifier |
| FW11-SED-0-12-01-033121 | L34670-3 | D3-N-MeFOSA | 36.4 | UJ |
| FW11-SED-0-12-01-033121 | L34670-3 | D5-N-EtFOSA | 27.0 | UJ |
| FW11-SED-0-12-01-033121 | L34670-3 | d7-NMe-FOSE | 38.5 | UJ |
| FW11-SED-0-12-01-033121 | L34670-3 | d9-NEt-FOSE | 33.2 | UJ |
| FW12-SED-0-12-01-033121 | L34670-4 | D3-N-MeFOSA | 49.1 | UJ |
| FW12-SED-0-12-01-033121 | L34670-4 | D5-N-EtFOSA | 38.6 | UJ |
| FW13-SED-0-12-01-033121 | L34670-5 | D3-N-MeFOSA | 40.5 | UJ |
| FW13-SED-0-12-01-033121 | L34670-5 | D5-N-EtFOSA | 33.6 | UJ |
| FW14-SED-0-12-01-033121 | L34670-6 | D5-N-EtFOSA | 42.9 | UJ |
| FW16-SED-0-12-01-033121 | L34670-7 | 13C2-4:2 FTS | 239 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW16-SED-0-12-01-033121 | L34670-7 | D3-N-MeFOSA | 42.6 | UJ |
| FW16-SED-0-12-01-033121 | L34670-7 | D5-N-EtFOSA | 32.3 | UJ |
| FW16-SED-0-12-01-033121 | L34670-7 | d7-NMe-FOSE | 49.2 | UJ |
| FW16-SED-0-12-01-033121 | L34670-7 | d9-NEt-FOSE | 45.0 | UJ |
| FW18-SED-0-12-01-033121 | L34670-8 | D3-N-MeFOSA | 33.4 | UJ |
| FW18-SED-0-12-01-033121 | L34670-8 | D5-N-EtFOSA | 24.4 | UJ |
| FW18-SED-0-12-01-033121 | L34670-8 | d7-NMe-FOSE | 46.6 | UJ |
| FW18-SED-0-12-01-033121 | L34670-8 | d9-NEt-FOSE | 39.7 | UJ |
| FW19-SED-0-12-01-033121 | L34670-9 | 13C2-4:2 FTS | 235 | No Qualifier |
| FW19-SED-0-12-01-033121 | L34670-9 | D3-N-MeFOSA | 30.1 | UJ |
| FW19-SED-0-12-01-033121 | L34670-9 | D5-N-EtFOSA | 23.8 | UJ |
| FW19-SED-0-12-01-033121 | L34670-9 | d7-NMe-FOSE | 39.5 | UJ |
| FW19-SED-0-12-01-033121 | L34670-9 | d9-NEt-FOSE | 31.9 | UJ |
| FW10-SED-0-12-03-033121 | L34670-10 | 13C2-4:2 FTS | 214 | No Qualifier |
| FW10-SED-0-12-03-033121 | L34670-10 | D3-N-MeFOSA | 33.0 | UJ |
| FW10-SED-0-12-03-033121 | L34670-10 | D5-N-EtFOSA | 25.1 | UJ |
| FW10-SED-0-12-03-033121 | L34670-10 | d7-NMe-FOSE | 44.6 | UJ |
| FW10-SED-0-12-03-033121 | L34670-10 | d9-NEt-FOSE | 37.8 | UJ |
| FW19-SED-0-12-02-033121 | L34670-11 | D3-N-MeFOSA | 40.1 | UJ |
| FW19-SED-0-12-02-033121 | L34670-11 | D5-N-EtFOSA | 28.6 | UJ |
| FW19-SED-0-12-02-033121 | L34670-11 | d7-NMe-FOSE | 49.7 | UJ |
| FW19-SED-0-12-02-033121 | L34670-11 | d9-NEt-FOSE | 45.2 | UJ |
| FW01-SED-0-12-01-040621 | L34747-1 | D3-N-MeFOSA | 34.4 | J+ |
| FW01-SED-0-12-01-040621 | L34747-1 | D5-N-EtFOSA | 27.3 | J+ |
| FW01-SED-0-12-01-040621 | L34747-1 | d7-NMe-FOSE | 47.9 | UJ |
| FW01-SED-0-12-01-040621 | L34747-1 | d9-NEt-FOSE | 39.6 | UJ |
| FW06-SED-0-12-01-040621 | L34747-2 | D3-N-MeFOSA | 37.9 | J+ |
| FW06-SED-0-12-01-040621 | L34747-2 | D5-N-EtFOSA | 29.4 | J+ |
| FW06-SED-0-12-01-040621 | L34747-2 | d9-NEt-FOSE | 44.8 | J+ |
| FW07-SED-0-24-01-040621 | L34747-3 | D3-N-MeFOSA | 30.0 | J+ |
| FW07-SED-0-24-01-040621 | L34747-3 | D5-N-EtFOSA | 21.9 | J+ |
| FW07-SED-0-24-01-040621 | L34747-3 | d9-NEt-FOSE | 44.3 | UJ |
| FW17-SED-0-12-01-040621 | L34747-4 | D5-N-EtFOSA | 40.6 | UJ |
| PW14-SED-0-12-01-040621 | L34747-5 | D5-N-EtFOSA | 42.3 | UJ |
| PW15-SED-0-12-01-040621 | L34747-6 | D3-N-MeFOSA | 48.7 | J+ |
| PW15-SED-0-12-01-040621 | L34747-6 | D5-N-EtFOSA | 37.0 | UJ |
| PW14-SED-0-12-02-040621 | L34747-7 | D5-N-EtFOSA | 40.5 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **FW19-SED-0-12-01-033121/FW19-SED-0-12-02-033121:** | | | |
| PFPeA | 0.376 | 0.139 J | >±RL |
| PFDA | 0.257 | 0.149 J | 53.2 |
| PFBA | 2.96 | 0.957 | 102.3 |
| PFHpA | 0.534 | 0.3 | 56.1 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| FW10-SED-0-12-01-033121 | L34670-2 | PFDoA |
| FW12-SED-0-12-01-033121 | L34670-4 | N-EtFOSAA |
| FW13-SED-0-12-01-033121 | L34670-5 | PFBS |
| FW14-SED-0-12-01-033121 | L34670-6 | N-EtFOSAA |
| FW16-SED-0-12-01-033121 | L34670-7 | PFDoA |
| FW01-SED-0-12-01-040621 | L34747-1 | PFHxA |
| FW07-SED-0-24-01-040621 | L34747-3 | N-EtFOSA |
| FW07-SED-0-24-01-040621 | L34747-3 | PFDoS |
| PW15-SED-0-12-01-040621 | L34747-6 | N-EtFOSAA |
| PW15-SED-0-12-01-040621 | L34747-6 | PFNA |
| PW14-SED-0-12-02-040621 | L34747-7 | PFTeDA |

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76616 Report date (mm/dd/yyyy): 6/14/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/13/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at elevated temperatures of 7.4 and 14.1 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Associated equipment blanks reported in WG76353:<br>EQ-LINER-01-040621 (L34749-1)<br>EQ-CATCHER-01-040621 (L34749-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | FW10-SED-0-12-03-033121 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%<br>PFOS 870%/957%<br>The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD ok<br>FDs: FW10-SED-0-12-01-033121/FW10-SED-0-12-03-033121<br>FW19-SED-0-12-01-033121/FW19-SED-0-12-02-033121<br>PW14-SED-0-12-01-040621/PW14-SED-0-12- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 02-040621 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/13/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76718 (WG76386)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/15/21 14:50 | NFDHA | 67.2% | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 5/15/21 17:01 | NFDHA | 46.3% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW02-SED-0-12-01-041421 | L34797-1 | D3-N-MeFOSA | 36.2 | J+ |
| FW02-SED-0-12-01-041421 | L34797-1 | D5-N-EtFOSA | 27.6 | UJ |
| FW02-SED-0-12-01-041421 | L34797-1 | d7-NMe-FOSE | 46.2 | UJ |
| FW02-SED-0-12-01-041421 | L34797-1 | d9-NEt-FOSE | 41.6 | UJ |
| FW03-SED-0-12-01-041421 | L34797-2 | D3-N-MeFOSA | 35.3 | J+ |
| FW03-SED-0-12-01-041421 | L34797-2 | D5-N-EtFOSA | 25.1 | UJ |
| FW03-SED-0-12-01-041421 | L34797-2 | d7-NMe-FOSE | 47.9 | UJ |
| FW03-SED-0-12-01-041421 | L34797-2 | d9-NEt-FOSE | 41.3 | UJ |
| FW04-SED-0-18-01-041621 | L34797-3 | 13C2-4:2 FTS | 205 | No Qualifier |
| FW04-SED-0-18-01-041621 | L34797-3 | D3-N-MeFOSA | 24.3 | J+ |
| FW04-SED-0-18-01-041621 | L34797-3 | D5-N-EtFOSA | 18 | R |
| FW04-SED-0-18-01-041621 | L34797-3 | d7-NMe-FOSE | 35.2 | UJ |
| FW04-SED-0-18-01-041621 | L34797-3 | d9-NEt-FOSE | 28.4 | UJ |
| FW04-SED-0-18-02-041621 | L34797-4 | D3-N-MeFOSA | 36.1 | J+ |
| FW04-SED-0-18-02-041621 | L34797-4 | D5-N-EtFOSA | 28.6 | J+ |
| FW04-SED-0-18-02-041621 | L34797-4 | d7-NMe-FOSE | 44.7 | UJ |
| FW04-SED-0-18-02-041621 | L34797-4 | d9-NEt-FOSE | 42.9 | UJ |
| FW05-SED-0-12-01-041421 | L34797-5 | D3-N-MeFOSA | 38.7 | J+ |
| FW05-SED-0-12-01-041421 | L34797-5 | D5-N-EtFOSA | 28.3 | J+ |
| FW05-SED-0-12-01-041421 | L34797-5 | d9-NEt-FOSE | 45.6 | UJ |
| FW08-SED-0-12-01-041521 | L34797-6 | D3-N-MeFOSA | 38.3 | J+ |
| FW08-SED-0-12-01-041521 | L34797-6 | D5-N-EtFOSA | 28.6 | J+ |
| FW08-SED-0-12-01-041521 | L34797-6 | d9-NEt-FOSE | 48.3 | UJ |
| FW08-SED-0-12-02-041521 | L34797-7 | D3-N-MeFOSA | 41.1 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW08-SED-0-12-02-041521 | L34797-7 | D5-N-EtFOSA | 30.2 | J+ |
| FW08-SED-0-12-02-041521 | L34797-7 | d9-NEt-FOSE | 48.6 | UJ |
| FW15-SED-0-24-01-041421 | L34797-8 | D3-N-MeFOSA | 41.7 | J+ |
| FW15-SED-0-24-01-041421 | L34797-8 | D5-N-EtFOSA | 34.3 | J+ |
| PW16-SED-0-12-01-041421 | L34797-9 | D3-N-MeFOSA | 49.3 | J+ |
| PW16-SED-0-12-01-041421 | L34797-9 | D5-N-EtFOSA | 41.3 | UJ |
| RC7P-SED-0-12-01-041521 | L34797-10 | D5-N-EtFOSA | 40.8 | UJ |
| RC7P-SED-0-12-03-041521 | L34797-11(A) | 13C2-4:2 FTS | 215 | No Qualifier |
| RC7AP-SED-0-24-01-041521 | L34797-12 | D3-N-MeFOSA | 47.1 | UJ |
| RC7AP-SED-0-24-01-041521 | L34797-12 | D5-N-EtFOSA | 37.6 | UJ |
| MW14C-SOIL-COMP-15-20-01-041921 | L34825-1 | D3-N-MeFOSA | 48.6 | UJ |
| MW14C-SOIL-COMP-15-20-01-041921 | L34825-1 | D5-N-EtFOSA | 46.4 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **FW04-SED-0-18-01-041621/FW04-SED-0-18-02-041621:** | | | |
| PFOS | 253 | 85.3 | 99.1 |
| PFUnA | 0.532 | 0.177 J | >±RL |
| N-EtFOSAA | 13.4 | 3.99 | 108.2 |
| PFHxA | 0.362 J | 0.193 | >±RL |
| PFDoA | 1.03 | 0.245 | >±RL |
| N-MeFOSA | 0.821 | 0.374 | >±RL |
| PFOA | 4.05 | 1.83 | 75.5 |
| PFDA | 1.3 | 0.553 | 80.6 |
| PFDS | 4.33 | 0.999 | 125 |
| PFHxS | 0.931 | 0.372 | >±RL |
| PFHpS | 0.537 | 0.222 | >±RL |
| N-EtFOSA | 0.892 J | 0.355 J | >±RL |
| PFNS | 0.901 | 0.331 | >±RL |
| PFOSA | 11.4 | 4.19 | 92.5 |
| PFDoS | 0.476 | 0.166 J | 96.6 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| FW02-SED-0-12-01-041421 | L34797-1 | PFDoS |
| FW04-SED-0-18-01-041621 | L34797-3 | N-EtFOSA |
| FW04-SED-0-18-02-041621 | L34797-4 | N-EtFOSA |
| FW05-SED-0-12-01-041421 | L34797-5 | N-EtFOSA |
| FW05-SED-0-12-01-041421 | L34797-5 | N-MeFOSAA |
| FW08-SED-0-12-01-041521 | L34797-6 | N-EtFOSA |
| FW08-SED-0-12-02-041521 | L34797-7 | N-EtFOSA |
| FW15-SED-0-24-01-041421 | L34797-8 | N-EtFOSA |
| FW15-SED-0-24-01-041421 | L34797-8 | PFDoA |
| PW16-SED-0-12-01-041421 | L34797-9 | N-MeFOSAA |
| RC7P-SED-0-12-01-041521 | L34797-10 | N-EtFOSAA |
| RC7P-SED-0-12-03-041521 | L34797-11 (A) | N-EtFOSAA |
| RC7P-SED-0-12-03-041521 | L34797-11 (A) | PFDoA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76718 Report date (mm/dd/yyyy): 5/27/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/17/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | The associated equipment blank was reported in WG76353:<br>EQ-SHOVEL-01-041621 (L34798-11)<br>The associated trip blank was reported in WG76392:<br>786425524905 (L34827-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | EQ-SHOVEL-01-041621:<br>6:2 FTS 1.95 J ng/L<br>786425524905 (Trip Blank) was nondetect |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | Sediment sample results were nondetect. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC7P-SED-0-12-03-041521 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%<br>Recoveries ok |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: NFDHA RPD 60.4% (ND - no qualifiers)<br>FDs: FW04-SED-0-18-01-041621/FW04-SED-0-18-02-041621<br>FW08-SED-0-12-01-041521/FW08-SED-0-12- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 02-041521<br>RC7P-SED-0-12-01-041521/RC7P-SED-0-12-03-041521 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/17/2021

**SGS AXYS DPWG76733** 9/3/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | | | |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW13D-GW-15-25-01-041321 | PFOS | 19271 | 25379 | 1 | 11.91 | 11.9 | 0.513 | 0.004 | L34798-12 |
| MW2B-GW-57-62-01-041421 | PFOS | 1102196 | 21613 | 1 | 829.30 | 829 | 0.495 | 0.004 | L34798-14 |
| LCS ( WG76389-102) | PFOS | 13613 | 22018 | 1 | 4.98 | 4.98 | 1.00 | 0.004 | WG76389-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 5/12/21 3:26 | 24577 | 38364 | 5.157 | 5.16 | 5.00 | 103.14 | 103% |
| CCV 5/12/21 9:19 | 23811 | 38889 | 4.929 | 4.93 | 5.00 | 98.57 | 98.6% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG76392-101): | PFOS | 0.050 | <0.4 | verified <0.4 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76733 (Analysis WG76389) dated 5/31/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 9/4/2021 |

The samples listed below were collected at the Project 1007 Site on April 13 through 16, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW2B-GW-57-62-01-041421 | | 4/14/2021 | L34798-14 |
| MW2C-GW-35-40-01-041321 | | 4/13/2021 | L34798-15 |
| MW2D-GW-7-17-01-041321 | | 4/13/2021 | L34798-16 |
| MW6D-GW-8-18-01-041321 | | 4/13/2021 | L34798-17 |
| MW6C-GW-35-40-02-041321 | Field Duplicate of MW2C-GW-35-40-01-041321 | 4/13/2021 | L34798-18 |
| MW6C-GW-35-40-03-041321 | Field Duplicate of MW2C-GW-35-40-01-041321; MS/MSD | 4/13/2021 | L34798-19 (A) |
| MW6C-GW-35-40-01-041321 | | 4/13/2021 | L34798-20 |
| MW13D-GW-15-25-01-041321 | | 4/13/2021 | L34798-12 |
| MW13C-GW-115-125-01-041421 | | 4/14/2021 | L34798-13 |
| **Field Quality Control Blanks:** | | | |
| EQ-DIPPER-01-041621 | Equipment Blank | 4/16/2021 | L34798-8 |
| EQ-BLADDER-01-041621 | Equipment Blank | 4/16/2021 | L34798-9 |
| EQ-PLATE-01-041621 | Equipment Blank | 4/16/2021 | L34798-10 |
| EQ-SHOVEL-01-041621 | Equipment Blank | 4/16/2021 | L34798-11 |
| 786161277646 | Trip Blank | 4/13/2021 | L34798-1 |
| 786161498570 | Trip Blank | 4/13/2021 | L34798-2 |
| 786161748459 | Trip Blank | 4/15/2021 | L34798-3 |
| 786162048000 | Trip Blank | 4/16/2021 | L34798-4 |
| 786159880329 | Trip Blank | 4/16/2021 | L34798-5 |
| 786160271056 | Trip Blank | 4/16/2021 | L34798-6 |
| 786160617288 | Trip Blank | 4/16/2021 | L34798-7 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and field blanks |
| ✗ | Labeled Compounds (Surrogates) |
| ✗ | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control sample (LCS) |
| ✗ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample receipt temperature, labeled compounds recoveries, matrix spikes, laboratory control samples, field duplicate imprecision and quantitations (estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact. Most of the samples were received above 6 °C as summarized in the table below. The reporting limits for nondetect results were qualified as estimated (UJ), and detects were qualified as estimated biased low (J-).

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| MW2B-GW-57-62-01-041421 | L34798-14 | 6.6 |
| MW2C-GW-35-40-01-041321 | L34798-15 | 6.6 |
| MW2D-GW-7-17-01-041321 | L34798-16 | 6.6 |
| MW6D-GW-8-18-01-041321 | L34798-17 | 6.6 |
| MW6C-GW-35-40-02-041321 | L34798-18 | 6.6 |
| MW6C-GW-35-40-03-041321 | L34798-19 (A) | 6.6 |
| MW6C-GW-35-40-01-041321 | L34798-20 | 6.6 |
| MW13D-GW-15-25-01-041321 | L34798-12 | 6.6 |
| MW13C-GW-115-125-01-041421 | L34798-13 | 6.6 |
| EQ-SHOVEL-01-041621 | L34798-11 | 4.5 |
| 786161277646 | L34798-1 | 6.6 |
| 786161498570 | L34798-2 | 6.5 |
| 786161748459 | L34798-3 | 7.9 |
| 786162048000 | L34798-4 | 7.4 |
| 786159880329 | L34798-5 | 6.4 |
| 786160271056 | L34798-6 | 7.3 |

The trip blank field IDs were updated to comply with MPCA chain-of-custody (CoC) requirements. Also, additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the two highest standards. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

NQ- Not quantitated

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Field Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Seven trip blanks were reported in this sample group. The trip blanks were nondetect, with the exception that 6:2 FTS was detected in trip blank 786160617288 (L34798-7) at a concentration of 2.47 J ng/L. The samples associated with this trip blank were reported in DPGG76747 (WG76392).

Four equipment blanks were reported in this sample group. 6:2 FTS was detected in EQ-SHOVEL-01-041621 (L34798-11) collected 4/16/21 at a concentration of 1.95 J ng/L. Sediment samples collected 4/16/21 were reported in DPWG76718 (WG76386) and associated 6:2 FTS results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| 786162048000 | L34798-4 | d9-NEt-FOSE | 44.9 | UJ |
| 786160271056 | L34798-6 | D3-N-MeFOSA | 49.3 | UJ |
| 786160271056 | L34798-6 | D5-N-EtFOSA | 49.5 | UJ |
| MW13D-GW-15-25-01-041321 | L34798-12 | 13C2-PFTeDA | 49.7 | UJ<br>PFTrDA: UJ |
| MW13D-GW-15-25-01-041321 | L34798-12 | D3-N-MeFOSA | 45.4 | UJ |
| MW13D-GW-15-25-01-041321 | L34798-12 | D5-N-EtFOSA | 40.5 | UJ |
| MW13D-GW-15-25-01-041321 | L34798-12 | d7-NMe-FOSE | 42.8 | UJ |
| MW13D-GW-15-25-01-041321 | L34798-12 | d9-NEt-FOSE | 37.9 | UJ |
| MW2C-GW-35-40-01-041321 | L34798-15 | 13C4-PFBA | 37.6 | J+ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was analyzed for sample MW6C-GW-35-40-03-041321 (L34798-19 (A)). MS/MSD recoveries and relative percent differences (RPDs) were within the MPCA control limits and were acceptable, except as indicated in bold in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **MW6C-GW-35-40-03-041321:** | | | |
| PFDoS | 105/**63.7** | 70-130 | This sample (and the associated field duplicates) were nondetect and qualified UJ:<br><br>MW6C-GW-35-40-01-041321 (L34798-20)<br>MW6C-GW-35-40-02-041321 (L34798-18)<br>MW6C-GW-35-40-03-041321 (L34798-19 (A)) |
| NFDHA | **57.9/64.2** | 70-130 | The associated results were nondetect and were qualified due to LCS recovery. |
| PFBA | **241/336** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **150/183** | 70-130 | |

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits, except as listed in the table below.

| LCS/LCSD IDs | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG76389-102 | NFDHA | 59.6 | 70-130 | Associated results were nondetect and were qualified UJ. |

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples MW6C-GW-35-40-02-041321 and MW6C-GW-35-40-03-041321 were collected as duplicates of MW6C-GW-35-40-01-041321. RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the reporting limit (RL), with the exception of those indicated in bold in the table below. Results associated with field imprecision were qualified as estimated (J).

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **RC3-WAT-BULK-01-091620-U/RC3-WAT-BULK-02-091620-U/RC3-WAT-BULK-03-091620-U:** | | | | |
| PFBA | 321 | 322 | 224 | **34** |
| PFPeA | 9.34 | 9.12 | 6.7 | **31** |

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| MW6C-GW-35-40-02-041321 | L34798-18 | PFDA | Yes | The ion ratio did not meet criteria. |
| MW13C-GW-115-125-01-041421 | L34798-13 | PFOS | Yes | The ion ratio did not meet criteria for one of the branched isomers. |
| MW13D-GW-15-25-01-041321 | L34798-12 | PFOS | Yes | Did not meet signal to noise ratio criteria for PFOS-I. |
| MW2B-GW-57-62-01-041421 | L34798-14 | PFOS | No | The PFOS result was qualified as estimated biased (J+) as it included quantitation of a branched isomer (PFOS-IV) that did not meet the ion ratio criteria. |
| MW2C-GW-35-40-01-041321 | L34798-15 | PFOS | No | |
| MW2D-GW-7-17-01-041321 | L34798-16 | PFOS | No | |
| MW6D-GW-8-18-01-041321 | L34798-17 | PFOS | No | |
| MW6C-GW-35-40-02-041321 | L34798-18 | PFOS | No | |
| MW6C-GW-35-40-03-041321 | L34798-19 (A) | PFOS | No | |
| MW6C-GW-35-40-01-041321 | L34798-20 | PFOS | No | |
| MW6C-GW-35-40-01-041321 | L34798-20 | PFHxS | No | The PFHxS result was qualified as estimated biased (J+) as it included quantitation of a branched isomer (PFHxS-III) that did not meet the ion ratio criteria. |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW2B-GW-57-62-01-041421 (L34798-14)<br>MW2C-GW-35-40-01-041321 (L34798-15)<br>MW2D-GW-7-17-01-041321 (L34798-16)<br>MW6D-GW-8-18-01-041321 (L34798-17)<br>MW6C-GW-35-40-02-041321 (L34798-18)<br>MW6C-GW-35-40-03-041321 (L34798-19 (A))<br>MW6C-GW-35-40-01-041321 (L34798-20)<br>MW13D-GW-15-25-01-041321 (L34798-12)<br>MW13C-GW-115-125-01-041421 (L34798-13)<br>EQ-SHOVEL-01-041621 (L34798-11)<br>786161277646 (L34798-1)<br>786161498570 (L34798-2)<br>786161748459 (L34798-3)<br>786162048000 (L34798-4)<br>786159880329 (L34798-5)<br>786160271056 (L34798-6) | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW13D-GW-15-25-01-041321 (L34798-12) | PFTeDA<br>PFTrDA<br>N-MeFOSA<br>N-EtFOSA<br>NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| MW2C-GW-35-40-01-041321 (L34798-15) | PFBA | ng/L | J+ | s |
| 786162048000 (L34798-4) | NEt-FOSE | ng/L | UJ | s |
| 786160271056 (L34798-6) | N-MeFOSA<br>N-EtFOSA | ng/L | UJ | s |
| MW6C-GW-35-40-01-041321 (L34798-20) | PFDoS | ng/L | UJ | m |
| MW6C-GW-35-40-02-041321 (L34798-18)<br>MW6C-GW-35-40-03-041321 (L34798-19 (A)) | NFDHA | ng/L | UJ | m |
| All samples | NFDHA | ng/L | UJ | l |
| MW6C-GW-35-40-01-041321 (L34798-20)<br>MW6C-GW-35-40-02-041321 (L34798-18)<br>MW6C-GW-35-40-03-041321 (L34798-19 (A)) | PFBA<br>PFPeA | ng/L | J | fd |
| MW6C-GW-35-40-02-041321 (L34798-18) | PFDA | ng/L | J | em |
| MW13D-GW-15-25-01-041321 (L34798-12)<br>MW13C-GW-115-125-01-041421 (L34798-13) | PFOS | ng/L | J | em |
| MW2B-GW-57-62-01-041421 (L34798-14)<br>MW2C-GW-35-40-01-041321 (L34798-15)<br>MW2D-GW-7-17-01-041321 (L34798-16)<br>MW6D-GW-8-18-01-041321 (L34798-17)<br>MW6C-GW-35-40-02-041321 (L34798-18)<br>MW6C-GW-35-40-03-041321 (L34798-19 (A))<br>MW6C-GW-35-40-01-041321 (L34798-20) | PFOS | ng/L | J+ | q |
| MW6C-GW-35-40-01-041321 (L34798-20) | PFHxS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

em Estimated maximum concentration
fd Field duplicate
l Laboratory control sample
m Matrix spike
q Quantitation
sr Sample receipt
s Surrogate (labeled compound)

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76737 (WG76353)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 7.4 °C (above the limit of ≤ 6 °C). Results reported for the samples were nondetects and qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/14/21 21:03 | NFDHA | 57.9% | 70-130% | NFDHA results were nondetect and previously qualified UJ due to sample receipt temperature. |
| CCV 5/15/21 3:08 | NFDHA | 50.0% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) as summarized below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EQ-CATCHER-01-040621 | L34749-2 | d9-NEt-FOSE | 49.5 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG76353-102 | NFDHA | 45.9 | 70-130 | NFDHA results were nondetect and previously qualified UJ due to sample receipt temperature. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76737 Report date (mm/dd/yyyy): 6/14/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/31/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at an elevated temperature of 7.4 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | Sample volume for MS/MSD not included. Only field QC blanks reported in this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Equipment blanks:<br>EQ-LINER-01-040621 (L34749-1)<br>EQ-CATCHER-01-040621 (L34749-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☐ | ☒ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/31/2021

**SGS AXYS DPWG76738** 9/6/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| RC3-WAT-BULK-01-030221 | PFOS | 79269 | 18898 | 1 | 61.39 | 61.4 | 0.550 | 0.004 | L34556-3 |
| WL6-WAT-BULK-01-030221 | PFOS | 70403 | 16381 | 1 | 65.15 | 65.2 | 0.531 | 0.004 | L34556-9 |
| LCS ( WG76389-102) | PFOS | 12527 | 21313 | 1 | 4.73 | 4.73 | 1.00 | 0.004 | WG76353-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 5/14/21 21:03 | 19371 | 33264 | 4.688 | 4.69 | 5.00 | 93.75 | 93.6% |
| CCV 5/15/21 3:08 | 20545 | 32363 | 5.110 | 5.11 | 5.00 | 102.20 | 102% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG76353-101): | PFOS | 0.125 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG76738 (Analysis WG76353) dated 6/14/2021 | |
| Analyses/Method: | SGS AXYS Method MLA-110 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 9/19/2021 |

The samples listed below were collected at the Project 1007 Site on March 2 and 8, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| RC3-WAT-BULK-01-030221 | | 3/2/2021 | L34556-3 |
| RC5-WAT-BULK-01-030221 | | 3/2/2021 | L34556-4 |
| RC5-WAT-BULK-02-030221 | Field Duplicate of RC5-WAT-BULK-01-030221 | 3/2/2021 | L34556-5 |
| RC5-WAT-BULK-03-030221 | Field Duplicate of RC5-WAT-BULK-01-030221; MS/MSD | 3/2/2021 | L34556-6 (A) |
| EP19-WAT-BULK-01-030221 | | 3/2/2021 | L34556-7 |
| EP16-WAT-BULK-01-030221 | | 3/2/2021 | L34556-8 |
| WL6-WAT-BULK-01-030221 | | 3/2/2021 | L34556-9 |
| WL15-WAT-BULK-01-030221 | | 3/2/2021 | L34556-10 |
| VB4-WAT-BULK-01-030221 | | 3/2/2021 | L34556-11 |
| VB4-WAT-BULK-02-030221 | Field Duplicate of VB4-WAT-BULK-01-030221 | 3/2/2021 | L34556-12 |
| VB5-WAT-BULK-01-030221 | | 3/2/2021 | L34556-13 |
| VB3-WAT-BULK-01-030221 | | 3/2/2021 | L34556-14 |
| VB7-WAT-BULK-01-030221 | | 3/2/2021 | L34556-15 |
| VB6-WAT-BULK-01-030221 | | 3/2/2021 | L34556-16 |
| RC3ST-WAT-BULK-01-030221 | | 3/2/2021 | L34556-17 |
| **Field Quality Control Blanks:** | | | |
| 784512055830 | Trip Blank | 3/8/2021 | L34556-1 |
| 784513589964 | Trip Blank | 3/8/2021 | L34556-2 |
| EQ-DIPPER-01-030821 | Equipment Blank | 3/8/2021 | L34556-18 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

- ✓ Data completeness (chain-of-custody (COC)/sample integrity
- ✓ Sample Receipt and Holding times
- ✗ Initial calibration/continuing calibration verification
- ✓ Laboratory and field blanks
- ✗ Labeled Compounds (Surrogates)
- ✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
- ✗ Laboratory control sample (LCS)
- ✗ Field duplicates
- ✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to labeled compounds recoveries, matrix spikes, laboratory control samples, field duplicate imprecision and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact, and the samples were received within the 6 °C temperature requirement.

The trip blank field IDs were updated to comply with MPCA chain-of-custody (CoC) requirements. In addition, an email included with the sample receipt information indicates the sample ID for RC3ST-WAT-BULK-01-030221 (L34556-17) should be RCST-WAT-BULK-01-030221.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

## Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the two highest standards. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/14/21 21:03 | NFDHA | 57.9% | 70-130% | NFDHA results were nondetect and qualified as estimated (UJ). |
| CCV 5/15/21 3:08 | NFDHA | 50.0% | 70-130% | |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Field Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Two trip blanks were reported in this sample group. The trip blank results were nondetect.

One equipment blank was reported in this sample group. The equipment blank was nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| 784512055830 | L34556-1 | 13C2-4:2 FTS | 220 | No Qualifiers |
| 784513589964 | L34556-2 | 13C2-4:2 FTS | 211 | No Qualifiers |
| RC3-WAT-BULK-01-030221 | L34556-3 | 13C2-4:2 FTS | 273 | No Qualifiers |
| RC5-WAT-BULK-01-030221 | L34556-4 | 13C2-4:2 FTS | 293 | No Qualifiers |
| RC5-WAT-BULK-02-030221 | L34556-5 | 13C2-4:2 FTS | 276 | No Qualifiers |
| RC5-WAT-BULK-02-030221 | L34556-5 | d9-NEt-FOSE | 45.9 | UJ |
| RC5-WAT-BULK-03-030221 | L34556-6 (A) | 13C2-4:2 FTS | 308 | No Qualifiers |
| RC5-WAT-BULK-03-030221 | L34556-6 (A) | 13C2-6:2 FTS | 202 | No Qualifiers |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C5-PFPeA | 44.7 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C5-PFHxA | 42.8 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C4-PFHpA | 40.8 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C8-PFOA | 35.8 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C9-PFNA | 35.2 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C6-PFDA | 35.5 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C7-PFUnA | 39 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C2-PFDoA | 42.8 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C2-PFTeDA | 39.2 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C3-PFBS | 30.9 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C3-PFHxS | 29 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C8-PFOS | 28.5 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C2-8:2 FTS | 49.4 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C8-PFOSA | 37 | J+ |
| EP19-WAT-BULK-01-030221 | L34556-7 | D3-N-MeFOSA | 28.2 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | D5-N-EtFOSA | 29.8 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | D3-MeFOSAA | 37.4 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | D5-EtFOSAA | 42.3 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | d7-NMe-FOSE | 26.6 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | d9-NEt-FOSE | 27.9 | UJ |
| EP19-WAT-BULK-01-030221 | L34556-7 | 13C3-HFPO-DA | 41.7 | UJ |
| EP16-WAT-BULK-01-030221 | L34556-8 | 13C2-4:2 FTS | 234 | No Qualifiers |
| WL6-WAT-BULK-01-030221 | L34556-9 | 13C2-4:2 FTS | 228 | No Qualifiers |
| WL6-WAT-BULK-01-030221 | L34556-9 | d9-NEt-FOSE | 47.1 | UJ |

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15-WAT-BULK-01-030221 | L34556-10 | 13C2-4:2 FTS | 219 | No Qualifiers |
| WL15-WAT-BULK-01-030221 | L34556-10 | d9-NEt-FOSE | 49.5 | UJ |
| VB4-WAT-BULK-01-030221 | L34556-11 | d9-NEt-FOSE | 46.3 | UJ |
| VB4-WAT-BULK-02-030221 | L34556-12 | 13C2-4:2 FTS | 217 | No Qualifiers |
| VB4-WAT-BULK-02-030221 | L34556-12 | d9-NEt-FOSE | 44.6 | UJ |
| VB5-WAT-BULK-01-030221 | L34556-13 | D5-N-EtFOSA | 48.7 | UJ |
| VB5-WAT-BULK-01-030221 | L34556-13 | d7-NMe-FOSE | 45.9 | UJ |
| VB5-WAT-BULK-01-030221 | L34556-13 | d9-NEt-FOSE | 39.2 | UJ |
| VB3-WAT-BULK-01-030221 | L34556-14 | d9-NEt-FOSE | 42.4 | UJ |
| VB7-WAT-BULK-01-030221 | L34556-15 | d9-NEt-FOSE | 49.1 | UJ |
| VB6-WAT-BULK-01-030221 | L34556-16 | d9-NEt-FOSE | 42.6 | UJ |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | 13C2-PFTeDA | 47.4 | UJ |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | 13C2-4:2 FTS | 346 | No Qualifiers |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | 13C2-6:2 FTS | 266 | No Qualifiers |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | d9-NEt-FOSE | 41.8 | UJ |
| EQ-DIPPER-01-030821 | L34556-18 | 13C2-4:2 FTS | 209 | No Qualifiers |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was analyzed for sample RC5-WAT-BULK-03-030221 (L34556-6 (A)). MS/MSD recoveries and RPDs were within the MPCA control limits, and were acceptable except as noted in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **RC5-WAT-BULK-03-030221:** | | | |
| NFDHA | **41.7/58.8 (RPD 34.1%)** | 70-130 | The associated results were nondetect and were previously qualified due to LCS recovery. |
| PFHpA | **61.6/37.1** | 70-130 | This sample (and the associated field duplicates) were nondetect and qualified UJ:<br><br>RC5-WAT-BULK-01-030221 (L34556-4)<br>RC5-WAT-BULK-02-030221 (L34556-5)<br>RC5-WAT-BULK-03-030221 (L34556-6 (A)) |
| PFBA | **276/NR** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFHxA | 71.1**/NR** | 70-130 | |
| PFOA | **NR/NR** | 70-130 | |
| PFOS | **255/474** | 70-130 | |

NR – Not reported

## LCS Results

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits, except as listed in the table below.

| LCS ID | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG76353-102 | NFDHA | 45.9 | 70-130 | Associated results were nondetect and were qualified UJ. |

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples RC5-WAT-BULK-02-030221 and RC5-WAT-BULK-03-030221 were collected as duplicates of RC5-WAT-BULK-01-030221; and sample VB4-WAT-BULK-02-030221 was collected as a field duplicate of VB4-WAT-BULK-01-030221. RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the RL, with the exception of those indicated in bold in the table below. Results associated with field imprecision were qualified as estimated (J).

| Analyte | Parent Sample Result (ug/L) | Field Duplicate Result (ug/L) | RPD (%) |
|---|---|---|---|
| **VB4-WAT-BULK-01-030221 / VB4-WAT-BULK-02-030221:** | | | |
| PFBA | 0.317 | 0.173 | **58.8** |
| PFBS | 0.00215 | 0.00121 J | **> ±RL** |
| PFHxA | 0.00299 | 0.00173 | **53.4** |
| PFPeA | 0.0119 | 0.0064 | **60.1** |

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were reviewed. The sample volumes listed in the raw instrument data do not match the sample size listed on the laboratory report or extraction log for sample L34556-6 through -18. The Audit Change Reports documents this discrepancy. In addition, the laboratory was requested to review the results and confirm the correct results were reported.

The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| RC5-WAT-BULK-02-030221 | L34556-5 | MeFOSAA | Yes | The ion ratio did not meet criteria (J+). |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | PFBS | Yes | The ion ratio and signal to noise criteria were not met (J+). |
| RC3-WAT-BULK-01-030221<br>RC5-WAT-BULK-01-030221<br>RC5-WAT-BULK-02-030221<br>RC5-WAT-BULK-03-030221<br>EP19-WAT-BULK-01-030221 | L34556-3<br>L34556-4<br>L34556-5<br>L34556-6 (A)<br>L34556-7 | PFOS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak (one or more) that did not meet the ion ratio criteria. |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| EP16-WAT-BULK-01-030221<br>WL6-WAT-BULK-01-030221<br>WL15-WAT-BULK-01-030221<br>VB3-WAT-BULK-01-030221<br>VB7-WAT-BULK-01-030221<br>RC3ST-WAT-BULK-01-030221 | L34556-8<br>L34556-9<br>L34556-10<br>L34556-14<br>L34556-15<br>L34556-17 | PFOS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak (one or more) that did not meet the ion ratio criteria. |
| RC5-WAT-BULK-01-030221<br>RC5-WAT-BULK-02-030221 | L34556-4<br>L34556-5 | EtFOSAA | No | |
| WL6-WAT-BULK-01-030221 | L34556-9 | PFOSA | No | |
| RC5-WAT-BULK-03-030221 | L34556-6 (A) | ETFOSAA | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak that did not meet the signal to noise criteria. |
| WL15-WAT-BULK-01-030221 | L34556-10 | PFOSA | No | |
| RC3ST-WAT-BULK-01-030221 | L34556-17 | PFHxA<br>PFDA<br>PFOSA | No | |
| VB7-WAT-BULK-01-030221 | L34556-15 | PFHxS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak that did not meet the ion ratio criteria, and a peak that did not meet the signal to noise criteria. |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| RC5-WAT-BULK-02-030221 (L34556-5)<br>WL6-WAT-BULK-01-030221 (L34556-9)<br>WL15-WAT-BULK-01-030221 (L34556-10)<br>VB4-WAT-BULK-01-030221 (L34556-11)<br>VB4-WAT-BULK-02-030221 (L34556-12)<br>VB5-WAT-BULK-01-030221 (L34556-13)<br>VB3-WAT-BULK-01-030221 (L34556-14)<br>VB7-WAT-BULK-01-030221 (L34556-15)<br>VB6-WAT-BULK-01-030221 (L34556-16)<br>RC3ST-WAT-BULK-01-030221 (L34556-17) | NEt-FOSE | ng/L | UJ | s |
| VB5-WAT-BULK-01-030221 (L34556-13) | N-EtFOSA | ng/L | UJ | s |
| RC3ST-WAT-BULK-01-030221 (L34556-17) | PFTeDA | ng/L | UJ | s |
| EP19-WAT-BULK-01-030221 (L34556-7) | PFBS<br>PFDA<br>PFHpA<br>PFHxA<br>PFHxS<br>PFNA<br>PFOA<br>PFOS<br>PFOSA<br>PFPeA | ng/L | J+ | s |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| EP19-WAT-BULK-01-030221 (L34556-7) | 8:2 FTS<br>EtFOSAA<br>HFPO-DA<br>MeFOSAA<br>N-EtFOSA<br>NEt-FOSE<br>N-MeFOSA<br>NMe-FOSE<br>PFDoA<br>PFTeDA<br>PFUnA | ng/L | UJ | s |
| RC5-WAT-BULK-01-030221 (L34556-4)<br>RC5-WAT-BULK-02-030221 (L34556-5)<br>RC5-WAT-BULK-03-030221 (L34556-6 (A)) | PFHpA | ng/L | J- | m |
| | NFDHA | ng/L | UJ | m |
| All samples | NFDHA | ng/L | UJ | c,l |
| VB4-WAT-BULK-01-030221 (L34556-11)<br>VB4-WAT-BULK-02-030221 (L34556-12) | PFBA<br>PFBS<br>PFHxA<br>PFPeA | ng/L | J | fd |
| RC5-WAT-BULK-02-030221 (L34556-5) | MeFOSAA | ng/L | J+ | em |
| RC3ST-WAT-BULK-01-030221 (L34556-17) | PFBS | ng/L | J+ | em |
| RC3-WAT-BULK-01-030221 (L34556-3)<br>RC5-WAT-BULK-01-030221 (L34556-4)<br>RC5-WAT-BULK-02-030221 (L34556-5)<br>RC5-WAT-BULK-03-030221 (L34556-6 (A))<br>EP19-WAT-BULK-01-030221 (L34556-7)<br>EP16-WAT-BULK-01-030221 (L34556-8)<br>WL6-WAT-BULK-01-030221 (L34556-9)<br>WL15-WAT-BULK-01-030221 (L34556-10)<br>VB3-WAT-BULK-01-030221 (L34556-14)<br>VB7-WAT-BULK-01-030221 (L34556-15)<br>RC3ST-WAT-BULK-01-030221 (L34556-17) | PFOS | ng/L | J+ | q |
| RC5-WAT-BULK-01-030221 (L34556-4)<br>RC5-WAT-BULK-02-030221 (L34556-5)<br>RC5-WAT-BULK-03-030221 (L34556-6 (A)) | EtFOSAA | ng/L | J+ | q |
| WL6-WAT-BULK-01-030221 (L34556-9)<br>WL15-WAT-BULK-01-030221 (L34556-10) | PFOSA | ng/L | J+ | q |
| RC3ST-WAT-BULK-01-030221 (L34556-17) | PFHxA<br>PFDA<br>PFOSA | ng/L | J+ | q |
| VB7-WAT-BULK-01-030221 (L34556-15) | PFHxS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

c calibration
em Estimated maximum concentration
fd Field duplicate
l Laboratory control sample
m Matrix spike
q Quantitation
s Surrogate (labeled compound)

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76740 (WG76351)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/11/21 17:38 | NFDHA | 45.3% | 70-130% | All NFDHA results were nondetect and associated with this closing CCV. NFDHA results were qualified UJ. |
| CCV 5/18/21 17:17 | NFDHA | 69.9% | 70-130% | NFDHA results were not associated with this CCV. |
| CCV 5/18/21 18:36 | ADONA | 144% | 70-130% | These analytes were not reported from this CCV. No qualifiers. |
| | 9Cl-PF3ONS | 146% | 70-130% | |
| | 11Cl-PF3OUdS | 145% | 70-130% | |
| | NFDHA | 33.0% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification. Three results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PW01-SED-0-12-01-031521 | L34605-1 | 13C2-4:2 FTS | 208 | No Qualifier |
| PW01-SED-0-12-01-031521 | L34605-1 | D3-N-MeFOSA | 22.7 | UJ |
| PW01-SED-0-12-01-031521 | L34605-1 | D5-N-EtFOSA | 15.4 | R |
| PW01-SED-0-12-01-031521 | L34605-1 | d7-NMe-FOSE | 33.6 | UJ |
| PW01-SED-0-12-01-031521 | L34605-1 | d9-NEt-FOSE | 27.6 | UJ |
| PW01-SED-0-12-02-031521 | L34605-2 | 13C2-4:2 FTS | 247 | No Qualifier |
| PW01-SED-0-12-02-031521 | L34605-2 | D3-N-MeFOSA | 22.3 | UJ |
| PW01-SED-0-12-02-031521 | L34605-2 | D5-N-EtFOSA | 14.3 | R |
| PW01-SED-0-12-02-031521 | L34605-2 | d7-NMe-FOSE | 34.2 | UJ |
| PW01-SED-0-12-02-031521 | L34605-2 | d9-NEt-FOSE | 27.4 | UJ |
| PW02-SED-0-24-01-031521 | L34605-3 | D3-N-MeFOSA | 44.8 | UJ |
| PW02-SED-0-24-01-031521 | L34605-3 | D5-N-EtFOSA | 35.6 | UJ |
| PW02-SED-0-24-01-031521 | L34605-3 | d9-NEt-FOSE | 48.4 | UJ |
| PW03-SED-0-12-01-031521 | L34605-4 | 13C2-PFTeDA | 39.0 | J+ |
| PW03-SED-0-12-01-031521 | L34605-4 | 13C2-4:2 FTS | 231 | No Qualifier |
| PW03-SED-0-12-01-031521 | L34605-4 | D3-N-MeFOSA | 25.4 | UJ |
| PW03-SED-0-12-01-031521 | L34605-4 | D5-N-EtFOSA | 16.8 | R |
| PW03-SED-0-12-01-031521 | L34605-4 | d7-NMe-FOSE | 34.5 | UJ |
| PW03-SED-0-12-01-031521 | L34605-4 | d9-NEt-FOSE | 27.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PW04-SED-0-12-01-031521 | L34605-5 | D3-N-MeFOSA | 28.8 | J+ |
| PW04-SED-0-12-01-031521 | L34605-5 | D5-N-EtFOSA | 21.0 | UJ |
| PW04-SED-0-12-01-031521 | L34605-5 | d7-NMe-FOSE | 37.3 | UJ |
| PW04-SED-0-12-01-031521 | L34605-5 | d9-NEt-FOSE | 30.5 | UJ |
| PW05-SED-0-12-01-031521 | L34605-6 | 13C2-4:2 FTS | 246 | No Qualifier |
| PW05-SED-0-12-01-031521 | L34605-6 | D3-N-MeFOSA | 30.3 | J+ |
| PW05-SED-0-12-01-031521 | L34605-6 | D5-N-EtFOSA | 21.5 | J+ |
| PW05-SED-0-12-01-031521 | L34605-6 | d7-NMe-FOSE | 40.3 | UJ |
| PW05-SED-0-12-01-031521 | L34605-6 | d9-NEt-FOSE | 32.9 | UJ |
| PW06-SED-0-12-01-031521 | L34605-7 | D3-N-MeFOSA | 45.3 | UJ |
| PW06-SED-0-12-01-031521 | L34605-7 | D5-N-EtFOSA | 36.6 | UJ |
| PW07-SED-0-12-01-031521 | L34605-8 | D3-N-MeFOSA | 35.8 | UJ |
| PW07-SED-0-12-01-031521 | L34605-8 | D5-N-EtFOSA | 25.7 | UJ |
| PW07-SED-0-12-01-031521 | L34605-8 | d7-NMe-FOSE | 45.7 | UJ |
| PW07-SED-0-12-01-031521 | L34605-8 | d9-NEt-FOSE | 38.9 | UJ |
| PW08-SED-0-12-01-031521 | L34605-9 | D3-N-MeFOSA | 31.8 | UJ |
| PW08-SED-0-12-01-031521 | L34605-9 | D5-N-EtFOSA | 24.0 | UJ |
| PW08-SED-0-12-01-031521 | L34605-9 | d7-NMe-FOSE | 46.6 | UJ |
| PW08-SED-0-12-01-031521 | L34605-9 | d9-NEt-FOSE | 41.1 | UJ |
| PW09-SED-0-12-01-031521 | L34605-10 | D3-N-MeFOSA | 40.6 | UJ |
| PW09-SED-0-12-01-031521 | L34605-10 | D5-N-EtFOSA | 31.7 | UJ |
| PW09-SED-0-12-01-031521 | L34605-10 | d9-NEt-FOSE | 48.1 | UJ |
| PW10-SED-0-12-01-031521 | L34605-11 | D3-N-MeFOSA | 32.7 | UJ |
| PW10-SED-0-12-01-031521 | L34605-11 | D5-N-EtFOSA | 24.4 | UJ |
| PW10-SED-0-12-01-031521 | L34605-11 | d7-NMe-FOSE | 46.5 | UJ |
| PW10-SED-0-12-01-031521 | L34605-11 | d9-NEt-FOSE | 41.8 | UJ |
| PW10-SED-0-12-02-031521 | L34605-12 | D3-N-MeFOSA | 31.7 | J+ |
| PW10-SED-0-12-02-031521 | L34605-12 | D5-N-EtFOSA | 23.2 | UJ |
| PW10-SED-0-12-02-031521 | L34605-12 | d7-NMe-FOSE | 47.1 | UJ |
| PW10-SED-0-12-02-031521 | L34605-12 | d9-NEt-FOSE | 41.1 | UJ |
| PW09-SED-0-12-03-031521 | L34605-13 (A) | D3-N-MeFOSA | 36.0 | UJ |
| PW09-SED-0-12-03-031521 | L34605-13 (A) | D5-N-EtFOSA | 28.1 | UJ |
| PW09-SED-0-12-03-031521 | L34605-13 (A) | d9-NEt-FOSE | 46.6 | UJ |
| PW11-SED-0-12-01-031521 | L34605-14 | D3-N-MeFOSA | 44.9 | UJ |
| PW11-SED-0-12-01-031521 | L34605-14 | D5-N-EtFOSA | 35.4 | UJ |
| PW12-SED-0-12-01-031521 | L34605-15 | 13C2-4:2 FTS | 268 | No Qualifier |
| PW12-SED-0-12-01-031521 | L34605-15 | D3-N-MeFOSA | 47.2 | UJ |
| PW12-SED-0-12-01-031521 | L34605-15 | D5-N-EtFOSA | 37.6 | UJ |
| PW13-SED-0-12-01-031521 | L34605-16 | 13C2-PFTeDA | 48.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PW13-SED-0-12-01-031521 | L34605-16 | 13C2-4:2 FTS | 234 | No Qualifier |
| PW13-SED-0-12-01-031521 | L34605-16 | D3-N-MeFOSA | 32.3 | UJ |
| PW13-SED-0-12-01-031521 | L34605-16 | D5-N-EtFOSA | 20.7 | UJ |
| PW13-SED-0-12-01-031521 | L34605-16 | d7-NMe-FOSE | 45.7 | UJ |
| PW13-SED-0-12-01-031521 | L34605-16 | d9-NEt-FOSE | 37.7 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (UJ/J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **PW10-SED-0-12-01-031521 / PW10-SED-0-12-02-031521:** | | | |
| 6:2 FTS | 0.137 U | 0.564 | >±RL |
| PFDA | 0.561 | 1.03 | 59 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PW01-SED-0-12-02-031521 | L34605-2 | N-MeFOSAA |
| PW02-SED-0-24-01-031521 | L34605-3 | N-EtFOSAA |
| PW04-SED-0-12-01-031521 | L34605-5 | N-MeFOSA |
| PW05-SED-0-12-01-031521 | L34605-6 | N-EtFOSA |
| PW06-SED-0-12-01-031521 | L34605-7 | PFDoA |
| PW07-SED-0-12-01-031521 | L34605-8 | N-EtFOSAA |
| PW10-SED-0-12-01-031521 | L34605-11 | N-MeFOSAA |
| PW10-SED-0-12-02-031521 | L34605-12 | N-MeFOSAA |
| PW09-SED-0-12-03-031521 | L34605-13 (A) | N-EtFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76740 Report date (mm/dd/yyyy): 5/31/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/7/2021

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | A QC sample (MS/MSD) was submitted with this SDG. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PW09-SED-0-12-03-031521 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%<br>NFDHA 49.1% (MS) - results previsouly qualified due to calibration. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD ok<br>FDs: PW01-SED-0-12-01-031521/PW01-SED-0-12-02-031521<br>PW10-SED-0-12-01-031521/PW10-SED-0-12-02-031521<br>PW09-SED-0-12-01-031521/PW09-SED-0-12-03-031521 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**Three results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/10/2021

**SGS AXYS DPWG76744** 9/25/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | | | |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| PZES-GW-6-11-01-041321 | PFOS | 584800 | 20368 | 1 | 445.31 | 445 | 0.519 | 0.004 | L34798-21 |
| PZBS-GW-6-11-01-041521 | PFOS | 35100 | 23454 | 1 | 23.26 | 23.3 | 0.518 | 0.004 | L34798-40 |
| LCS ( WG76390-102) | PFOS | 13855 | 24178 | 1 | 4.61 | 4.61 | 1.00 | 0.004 | WG76390-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 5/13/21 15:21 | 18661 | 31717 | 4.736 | 4.74 | 5.00 | 94.72 | 96.5% |
| CCV 5/15/21 3:08 | 20169 | 31122 | 5.217 | 5.22 | 5.00 | 104.33 | 104% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG76353-101): | PFOS | 0.052 | <0.4 | verified <0.4 |

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76744 (Analysis WG76390) dated 5/31/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 9/6/2021 |

The samples listed below were collected at the Project 1007 Site on April 13 through 15, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| PZES-GW-6-11-01-041321 | | 4/13/2021 | L34798-21 |
| PZES-GW-6-11-02-041321 | Field Duplicate of PZES-GW-6-11-01-041321 | 4/13/2021 | L34798-22 |
| PZES-GW-6-11-03-041321 | Field Duplicate of PZES-GW-6-11-01-041321; MS/MSD | 4/13/2021 | L34798-23 (A) |
| PZED-GW-16-21-01-041321 | | 4/13/2021 | L34798-24 |
| PZFD-GW-33-38-01-041321 | | 4/13/2021 | L34798-25 |
| PZFD-GW-33-38-02-041321 | Field Duplicate of PZFD-GW-33-38-01-041321 | 4/13/2021 | L34798-26 |
| PZFS-GW-18-28-01-041321 | | 4/13/2021 | L34798-28 |
| PZDS-GW-20-30-01-041421 | | 4/14/2021 | L34798-29 |
| PZDS-GW-20-30-02-041421 | Field Duplicate of PZDS-GW-20-30-01-041421 | 4/14/2021 | L34798-30 |
| PZDD-GW-35-40-01-041421 | | 4/14/2021 | L34798-31 |
| PZGS-GW-20-30-01-041421 | | 4/14/2021 | L34798-32 |
| PZGD-GW-35-40-01-041421 | | 4/14/2021 | L34798-33 |
| PZHS-GW-6-16-01-041421 | | 4/14/2021 | L34798-34 |
| PZHD-GW-21-26-01-041421 | | 4/14/2021 | L34798-35 |
| PZJS-GW-15-25-01-041421 | | 4/14/2021 | L34798-36 |
| PZJD-GW-30-35-01-041421 | | 4/14/2021 | L34798-37 |
| PZAS-GW-6-16-01-041521 | | 4/15/2021 | L34798-38 |
| PZAD-GW-21-26-01-041521 | | 4/15/2021 | L34798-39 |
| PZBS-GW-6-11-01-041521 | | 4/15/2021 | L34798-40 |
| PZBD-GW-16-21-01-041521 | | 4/15/2021 | L34798-41 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.

- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✗ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds (Surrogates) |
| ✗ | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| ✓ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample receipt temperature, labeled compounds recoveries, matrix spikes, and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact. The samples were received above 6 °C at temperatures of 6.5, 7.5, and 7.9 °C. The reporting limits for nondetect results were qualified as estimated (UJ), and detects were qualified as estimated biased low (J-).

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

## Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the two highest standards. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/13/21 15:21 | NFDHA | 55.6% | 70-130% | NFDHA results were nondetect and qualified as estimated (UJ). |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blanks associated with this sample set were reported in SDG DPWG76733 (WG76389) and were nondetect.

## Labeled Compounds

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZES-GW-6-11-01-041321 | L34798-21 | 13C2-4:2 FTS | 207 | No Qualifiers |
| PZES-GW-6-11-02-041321 | L34798-22 | 13C2-4:2 FTS | 230 | No Qualifiers |
| PZES-GW-6-11-03-041321 | L34798-23 (A) | 13C2-4:2 FTS | 225 | No Qualifiers |
| PZED-GW-16-21-01-041321 | L34798-24 | 13C2-4:2 FTS | 239 | No Qualifiers |
| PZJD-GW-30-35-01-041421 | L34798-37 | 13C4-PFBA | 35.2 | J+ |
| PZAS-GW-6-16-01-041521 | L34798-38 | 13C4-PFBA | 33.2 | J+ |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was analyzed for sample PZES-GW-6-11-03-041321 (L34798-23 (A)). MS/MSD recoveries and RPDs were within the MPCA control limits, and were acceptable except as noted in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **PZES-GW-6-11-03-041321:** | | | |
| NFDHA | **58.1/63.1** | 70-130 | This sample (and the associated field duplicates) were nondetect and qualified UJ:<br>PZES-GW-6-11-01-041321 (L34798-21)<br>PZES-GW-6-11-02-041321 (L34798-22)<br>PZES-GW-6-11-03-041321 (L34798-23 (A)) |
| PFHxA | **133/**72.4 | 70-130 | The associated results were qualified as estimated biased high (J+):<br>PZES-GW-6-11-01-041321 (L34798-21)<br>PZES-GW-6-11-02-041321 (L34798-22)<br>PZES-GW-6-11-03-041321 (L34798-23 (A)) |
| PFBA | **157/131** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **238/299** | 70-130 | |
| PFOS | **147/55.4** | 70-130 | |

## LCS Results

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits.

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples listed below were collected as field duplicates.

| Parent Sample | Field Duplicate | Triplicate Sample |
|---|---|---|
| PZES-GW-6-11-01-041321 (L34798-21) | PZES-GW-6-11-02-041321 (L34798-22) | PZES-GW-6-11-03-041321 (L34798-23 (A)) |
| PZFD-GW-33-38-01-041321 (L34798-25) | PZFD-GW-33-38-02-041321 (L34798-26) | -- |
| PZDS-GW-20-30-01-041421 (L34798-29) | PZDS-GW-20-30-02-041421 (L34798-30) | -- |

RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the reporting limit (RL).

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| PZGD-GW-35-40-01-041421 | L34798-33 | PFPeS | Yes | The ion ratio did not meet criteria, result qualified estimated biased high (J+). |
| PZAS-GW-6-16-01-041521 | L34798-38 | PFOS | Yes | The ion ratio did not meet criteria for two of the branched isomers, and result qualified estimated biased high (J+). |
| PZFD-GW-33-38-01-041321<br>PZFD-GW-33-38-02-041321 | L34798-25<br>L34798-26 | PFOS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak (one or more) that did not meet the ion ratio criteria. |
| PZES-GW-6-11-01-041321<br>PZES-GW-6-11-03-041321<br>PZED-GW-16-21-01-041321<br>PZFD-GW-33-38-01-041321<br>PZFD-GW-33-38-02-041321<br>PZFS-GW-18-28-01-041321<br>PZHS-GW-6-16-01-041421<br>PZHD-GW-21-26-01-041421 | L34798-21<br>L34798-23 (A)<br>L34798-24<br>L34798-25<br>L34798-26<br>L34798-28<br>L34798-34<br>L34798-35 | PFNA | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak that did not meet the signal to noise criteria. |
| PZJS-GW-15-25-01-041421<br>PZAS-GW-6-16-01-041521 | L34798-36<br>L34798-38 | PFOA | No | |
| PZJS-GW-15-25-01-041421<br>PZJD-GW-30-35-01-041421<br>PZBS-GW-6-11-01-041521 | L34798-36<br>L34798-37<br>L34798-40 | PFOS | No | |
| PZFS-GW-18-28-01-041321<br>PZJS-GW-15-25-01-041421 | L34798-28<br>L34798-36 | PFOSA | No | |
| PZJD-GW-30-35-01-041421<br>PZAD-GW-21-26-01-041521 | L34798-37<br>L34798-39 | PFHxS | No | |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All Samples | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| PZJD-GW-30-35-01-041421 (L34798-37)<br>PZAS-GW-6-16-01-041521 (L34798-38) | PFBA | ng/L | J+ | s |
| PZES-GW-6-11-01-041321 (L34798-21) | PFHxA | ng/L | J+ | m |
| PZES-GW-6-11-02-041321 (L34798-22)<br>PZES-GW-6-11-03-041321 (L34798-23 (A)) | NFDHA | ng/L | UJ | m |
| All samples | NFDHA | ng/L | UJ | c |
| PZGD-GW-35-40-01-041421 (L34798-33) | PFPeS | ng/L | J+ | em |
| PZAS-GW-6-16-01-041521 (L34798-38) | PFOS | ng/L | J+ | em |
| PZES-GW-6-11-01-041321 (L34798-21)<br>PZES-GW-6-11-03-041321 (L34798-23 (A))<br>PZED-GW-16-21-01-041321 (L34798-24)<br>PZFD-GW-33-38-01-041321 (L34798-25)<br>PZFD-GW-33-38-02-041321 (L34798-26)<br>PZFS-GW-18-28-01-041321 (L34798-28)<br>PZHS-GW-6-16-01-041421 (L34798-34)<br>PZHD-GW-21-26-01-041421 (L34798-35) | PFNA | ng/L | J+ | q |
| PZJS-GW-15-25-01-041421 (L34798-36)<br>PZAS-GW-6-16-01-041521 (L34798-38) | PFOA | ng/L | J+ | q |
| PZJS-GW-15-25-01-041421 (L34798-36)<br>PZJD-GW-30-35-01-041421 (L34798-37)<br>PZBS-GW-6-11-01-041521 (L34798-40) | PFOS | ng/L | J+ | q |
| PZFS-GW-18-28-01-041321 (L34798-28)<br>PZJS-GW-15-25-01-041421 (L34798-36) | PFOSA | ng/L | J+ | q |
| PZJD-GW-30-35-01-041421 (L34798-37)<br>PZAD-GW-21-26-01-041521 (L34798-39) | PFHxS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

- J Estimated (+/- indicates high or low bias)
- UJ Estimated reporting limit

(2): Reason Codes:

- c calibration
- em Estimated maximum concentration
- m Matrix spike
- q Quantitation
- sr Sample receipt
- s Surrogate (labeled compound)

# Attachment A

**SGS AXYS DPGG76747** 9/1/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form (p 150) | Page 558 |
| | | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 | 656 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 | 675 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 | 695 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 | 713 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 | 732 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 | 751 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 | 770 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 | 789 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 | 808 |
| | | Avg: | 1.2583 | 1.25 | |
| | | %RSD: | 5.2577 | 5.50 | |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| WL6-WAT-BULK-02-041621 | PFOS | 150917 | 19088 | 1 | 118.08 | 118 | 0.539 | 0.004 | L34798-61 |
| VB7-WAT-BULK-02-041621 | PFOS | 7692 | 21739 | 1 | 5.35 | 5.35 | 0.532 | 0.004 | L34798-69 |
| LCS (WG76392-102) | PFOS | 13944 | 21736 | 1 | 5.16 | 5.16 | 1.00 | 0.004 | WG76392-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 5/15/21 20:03 | 18153 | 30841 | 4.738 | 4.74 | 5.00 | 94.76 | 94.8% |
| CCV 5/16/21 1:04 | 17650 | 27064 | 5.250 | 5.25 | 5.00 | 104.99 | 105% |

| | | Raw Data (ng/L) | Reported (ng/L) | | |
|---|---|---|---|---|---|
| Method Blank ( WG76392-101): | PFOS | 0.066 | <0.4 | verified <0.4 | p 449 |

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# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPGG76747 (Analysis WG76392) dated 5/31/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 9/2/2021 |

The samples listed below were collected at the Project 1007 Site on April 16, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| WL6-WAT-BULK-02-041621 | Field Duplicate of WL6-WAT-BULK-01-041621 | 4/16/21 | L34798-61 |
| WL6-WAT-BULK-03-041621 | Field Duplicate of WL6-WAT-BULK-01-041621; MS/MSD | 4/16/21 | L34798-62 (A) |
| WL15-WAT-BULK-01-041621 | | 4/16/21 | L34798-63 |
| VB3-WAT-BULK-01-041621 | | 4/16/21 | L34798-64 |
| VB4-WAT-BULK-01-041621 | | 4/16/21 | L34798-65 |
| VB5-WAT-BULK-01-041621 | | 4/16/21 | L34798-66 |
| VB6-WAT-BULK-01-041621 | | 4/16/21 | L34798-67 |
| VB7-WAT-BULK-01-041621 | | 4/16/21 | L34798-68 |
| VB7-WAT-BULK-02-041621 | Field Duplicate of VB7-WAT-BULK-01-041621 | 4/16/21 | L34798-69 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✗ Sample Receipt and Holding times
✓ Initial calibration/continuing calibration verification
✓ Laboratory and field blanks

✗ Labeled Compounds
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓ Laboratory control sample (LCS)
✓ Field duplicates
✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of three PFBA results that were rejected (R) due to severely low labeled compound recoveries. In addition, NFDHA results were reported in the laboratory report as not quantifiable and values reported in the database were rejected (R). Three PFOSA results were qualified B as the sample concentrations were within 10 times the blank concentration. Additionally, some of the results were qualified as estimated (J/UJ) due to sample receipt temperature, labeled compound recoveries, matrix spike or quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact. Four of the samples were received above 6 °C. The reporting limit for nondetect results were qualified as estimated (UJ), and detects were qualified as estimated biased low (J-).

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the three highest standards. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

NQ- Not quantitated

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/15/21 20:03 | NFDHA | 41.2% | 70-130% | NFDHA results were qualified as not quantifiable (NQ) by the laboratory, and no result was reported. Results were rejected (R) in the database as discussed in the Quantitation and Results Verification section. |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Field Blanks**

Method blanks were analyzed to assess laboratory contamination. PFOSA was detected in the method blank at a concentration of 0.521 J ng/L. Associated sample detects were within 10 times the blank concentration and were qualified B.

Two trip blanks were associated with this sample group per CoC (L34798-6 and L34798-7 reported in DPWG76733/WG76389). The trip blank results were nondetect, with the exception that 6:2 FTS was

detected in trip blank 786160617288 (L34798-7) at a concentration of 2.47 J ng/L. 6:2 FTS was not detected in the associated samples.

Three equipment blanks were reported on the COC with the samples included in this SDG. These equipment blanks were reported in DPWG76733/WG76389 and the results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of the results listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification. Three results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL6-WAT-BULK-02-041621 | L34798-61 | 13C4-PFBA | 17.6 | R |
| WL6-WAT-BULK-02-041621 | L34798-61 | D5-N-EtFOSA | 48.2 | UJ |
| WL6-WAT-BULK-03-041621 | L34798-62 (A) | 13C4-PFBA | 17.5 | R |
| WL15-WAT-BULK-01-041621 | L34798-63 | 13C4-PFBA | 12.1 | R |
| WL15-WAT-BULK-01-041621 | L34798-63 | D5-N-EtFOSA | 45.5 | UJ |
| VB3-WAT-BULK-01-041621 | L34798-64 | 13C4-PFBA | 40.7 | J+ |
| VB3-WAT-BULK-01-041621 | L34798-64 | D5-N-EtFOSA | 49.4 | UJ |
| VB4-WAT-BULK-01-041621 | L34798-65 | D3-N-MeFOSA | 47.2 | UJ |
| VB4-WAT-BULK-01-041621 | L34798-65 | D5-N-EtFOSA | 49.2 | UJ |
| VB5-WAT-BULK-01-041621 | L34798-66 | D3-N-MeFOSA | 45.5 | UJ |
| VB5-WAT-BULK-01-041621 | L34798-66 | D5-N-EtFOSA | 44.0 | UJ |
| VB6-WAT-BULK-01-041621 | L34798-67 | D3-N-MeFOSA | 49.9 | UJ |
| VB6-WAT-BULK-01-041621 | L34798-67 | D5-N-EtFOSA | 47.1 | UJ |
| VB7-WAT-BULK-01-041621 | L34798-68 | 13C2-4:2 FTS | 201 | No Qualifier |
| VB7-WAT-BULK-01-041621 | L34798-68 | D3-N-MeFOSA | 48.9 | UJ |
| VB7-WAT-BULK-01-041621 | L34798-68 | D5-N-EtFOSA | 47.8 | UJ |
| VB7-WAT-BULK-02-041621 | L34798-69 | 13C2-4:2 FTS | 203 | No Qualifier |
| VB7-WAT-BULK-02-041621 | L34798-69 | D5-N-EtFOSA | 49.0 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was analyzed for sample WL6-WAT-BULK-03-041621 (L34798-62). MS/MSD recoveries and relative percent differences (RPDs) were within the MPCA control limits, and were acceptable except as noted in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **WL6-WAT-BULK-03-041621:** | | | |
| 3:3 FTCA | **23.5/24.2** | 70-130 | This sample (and the associated field duplicates) were nondetect and qualified UJ:<br><br>WL6-WAT-BULK-01-041621 (WG76391)<br>WL6-WAT-BULK-02-041621<br>WL6-WAT-BULK-03-041621 |
| PFMPA | **40.3/42.2** | 70-130 | The results for this sample (and the associated field duplicates) were qualified as estimated biased low (J-):<br><br>WL6-WAT-BULK-01-041621 (WG76391)<br>WL6-WAT-BULK-02-041621<br>WL6-WAT-BULK-03-041621 |
| PFBA | **-191/22.2** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **-46.8/-64.2** | 70-130 | |
| PFOS | **205/26.5** | 70-130 | |

## LCS Results

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits.

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples WL6-WAT-BULK-02-041621 and WL6-WAT-BULK-03-041621 were collected as duplicates of WL6-WAT-BULK-01-041621 (L34798-60; WG76391); and sample VB7-WAT-BULK-02-041621 was collected as a field duplicate of VB7-WAT-BULK-01-041621. RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the reporting limit (RL).

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| VB7-WAT-BULK-01-041621 | L34798-68 | PFOS | Yes | Did not meet signal to noise ratio criteria for PFOS peaks. Result was qualified as estimated (J). |
| WL6-WAT-BULK-02-041621 | L34798-61 | PFOS | No | The PFOS result was qualified as estimated biased (J+) as it |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| | | | | included quantitation of a branched isomer (PFOS-IV) that did not meet the ion ratio criteria. |

NFDHA results were qualified as not quantifiable (NQ) by the laboratory, as this compound did not adequately meet several method performance indicators. A value was not reported in the laboratory report and these results were rejected (R) in the database.

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| WL6-WAT-BULK-02-041621 (L34798-61)<br>WL6-WAT-BULK-03-041621 (L34798-62 (A))<br>WL15-WAT-BULK-01-041621 (L34798-63)<br>VB3-WAT-BULK-01-041621 (L34798-64) | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| WL6-WAT-BULK-02-041621 (L34798-61)<br>WL6-WAT-BULK-03-041621 (L34798-62 (A))<br>WL15-WAT-BULK-01-041621 (L34798-63) | PFOSA | ng/L | B | mb |
| VB4-WAT-BULK-01-041621 (L34798-65)<br>VB5-WAT-BULK-01-041621 (L34798-66)<br>VB6-WAT-BULK-01-041621 (L34798-67)<br>VB7-WAT-BULK-01-041621 (L34798-68) | EtFOSA<br>MeFOSA | ng/L | UJ | s |
| WL6-WAT-BULK-02-041621 (L34798-61)<br>WL15-WAT-BULK-01-041621 (L34798-63)<br>VB3-WAT-BULK-01-041621 (L34798-64)<br>VB7-WAT-BULK-02-041621 (L34798-69) | EtFOSA | ng/L | UJ | s |
| WL6-WAT-BULK-02-041621 (L34798-61)<br>WL6-WAT-BULK-03-041621 (L34798-62 (A))<br>WL15-WAT-BULK-01-041621 (L34798-63) | PFBA | ng/L | R | s |
| VB3-WAT-BULK-01-041621 (L34798-64) | PFBA | ng/L | J+ | s |
| WL6-WAT-BULK-01-041621 (L34798-60; WG76391)<br>WL6-WAT-BULK-02-041621 (L34798-61)<br>WL6-WAT-BULK-03-041621 (L34798-62 (A)) | 3:3 FTCA | ng/L | UJ | m |
| | PFMPA | ng/L | J- | m |
| VB7-WAT-BULK-01-041621 (L34798-68) | PFOS | ng/L | J | em |
| WL6-WAT-BULK-02-041621 (L34798-61) | PFOS | ng/L | J+ | q |
| All samples | NFDHA | ng/L | R | q |

(1): Data Validation Qualifiers:

- B Laboratory contamination
- J Estimated (+/- indicates high or low bias)
- R Rejected
- UJ Estimated reporting limit

(2): Reason Codes:

| | |
|---|---|
| em | Estimated maximum concentration |
| mb | Method blank |
| m | Matrix spike |
| q | Quantitation |
| sr | Sample receipt |
| s | Surrogate (labeled compound) |

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG76761 (WG76392)**

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperature greater than 6 °C. Associated results were nondetects and qualified UJ.

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| 786424205776 | L34827-1 | 7.4 |
| 786424857986 | L34827-2 | 8.8 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/15/21 20:03 | NFDHA | 41.2% | 70-130% | NFDHA results were qualified as not quantifiable (NQ) by the laboratory, and no results were reported. Results rejected (R) as discussed in Section 10. |

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| WG76392-101 | PFOSA | 0.521 J | Associated detects within 10 times the method blank concentration were qualified B:<br><br>RC21-WAT-BULK-01-042221 (L34827-4)<br>EP17-WAT-BULK-01-042221 (L34827-5)<br>EP19-WAT-BULK-01-042221 (L34827-6) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC21-WAT-BULK-01-042221 | L34827-4 | 13C4-PFBA | 20.6 | J+ |
| RC21-WAT-BULK-01-042221 | L34827-4 | 13C2-4:2 FTS | 208 | No Qualifiers |
| EP17-WAT-BULK-01-042221 | L34827-5 | 13C2-4:2 FTS | 232 | No Qualifiers |
| EP19-WAT-BULK-01-042221 | L34827-6 | 13C4-PFBA | 20.7 | J+ |
| EP19-WAT-BULK-01-042221 | L34827-6 | 13C2-4:2 FTS | 232 | No Qualifiers |

**10. Additional comments about lab report:** NFDHA results were qualified as not quantifiable (NQ) by the laboratory, as this compound did not adequately meet several method performance indicators. NFDHA values were not reported in the laboratory report; however, values were reported in the EDD and these results were rejected (R).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG76761 Report date (mm/dd/yyyy): 5/31/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/31/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | Two of the samples were received at elevated temperatures of 7.4 and 8.8 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | Sample volume for MS/MSD not included. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks:<br>786424205776 (L34827-1)<br>786424857986 (L34827-2)<br>786425524905 (L34827-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFOSA 0.521 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

**NFDHA results were reported as not quantifiable (NQ) by the laboratory. These results were rejected (R) in the database.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/31/2021

**SGS AXYS DPWG76830** 9/26/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW14A-GW-316-326-01-041621 | PFOS | 649136 | 25586 | 1 | 380.31 | 380 | 0.537 | 0.004 | L34828-1 |
| MW3B-GW-110-130-01-041921 | PFOS | 1058547 | 22925 | 1 | 687.03 | 687 | 0.541 | 0.004 | L34828-11 |
| LCS (WG76457-102 ) | PFOS | 15971 | 26365 | 1 | 4.88 | 4.88 | 1.00 | 0.004 | WG76457-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 5/25/21 21:00 | 24223 | 41163 | 4.737 | 4.74 | 5.00 | 94.74 | 96.4% |
| CCV 5/26/21 2:26 | 24531 | 40014 | 4.935 | 4.94 | 5.00 | 98.70 | 98.7% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG76353-101): | PFOS | 0.000 | <0.4 | verified <0.4 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76830 (Analysis WG76457) dated 6/7/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 9/26/2021 |

The samples listed below were collected at the Project 1007 Site on April 15 through 23, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW14A-GW-316-326-01-041621 | | 4/16/2021 | L34828-1 |
| MW14B-GW-60-70-01-041621 | | 4/16/2021 | L34828-2 |
| MW4A-GW-140-160-01-041521 | | 4/15/2021 | L34828-3 |
| MW4A-GW-140-160-02-041521 | Field Duplicate of MW4A-GW-140-160-01-041521 | 4/15/2021 | L34828-4 |
| MW1A-GW-360-370-01-041621 | | 4/16/2021 | L34828-5 |
| MW1C-GW-51-55-01-042121 | | 4/21/2021 | L34828-6 |
| MW1C-GW-40-45-01-042221 | | 4/22/2021 | L34828-7 |
| MW2A-GW-240-263-01-041921 | | 4/19/2021 | L34828-8 |
| MW17A-GW-230-240-01-041921 | | 4/19/2021 | L34828-9 |
| MW17B-GW-90-100-01-041921 | | 4/19/2021 | L34828-10 |
| MW3B-GW-110-130-01-041921 | | 4/19/2021 | L34828-11 |
| MW5B-GW-110-120-01-041921 | | 4/19/2021 | L34828-12 |
| MW5A-GW-210-220-01-041921 | | 4/19/2021 | L34828-13 |
| MW6A-GW-185-192-01-042121 | | 4/21/2021 | L34828-14 |
| MW6B-GW-140-150-01-041921 | | 4/19/2021 | L34828-15 |
| MW3A-GW-230-250-01-041921 | | 4/19/2021 | L34828-16 |
| MW9B-GW-90-100-01-042321 | | 4/23/2021 | L34828-17 |
| MW15B-GW-215-225-01-041921 | | 4/19/2021 | L34828-18 |
| MW15A-GW-330-340-01-041921 | | 4/19/2021 | L34828-19 |
| MW7A-GW-200-210-01-041921 | | 4/19/2021 | L34828-20 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✗ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control sample (LCS) |
| ✓ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of two results that were rejected (R) due to severely low labeled compound recoveries. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample receipt temperature, calibrations, labeled compounds recoveries, laboratory control samples, and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact. The samples were received above 6 °C at temperatures of 7.4 and 8.8 °C. The reporting limits for nondetect results were qualified as estimated (UJ), and detects were qualified as estimated biased low (J-).

Additional samples (not reported in this SDG) were listed on the COCs.

The login reports and email documentation indicate that there was a typo on the CoC for sample L34828-5. The sample was logged in as MW1A-GW-360-370-01-041621. In addition, sample L34828-20 was not listed on the CoC, and the laboratory requested confirmation of the sample ID as indicated in email documentation included in the laboratory report.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the two highest standards. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/25/21 21:00 | NFDHA | 57.6% | 70-130% | NFDHA results were nondetect and qualified as estimated (UJ). |
| CCV 5/26/21 2:26 | NFDHA | 63.2% | 70-130% | |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blanks associated with this sample set were reported in SDG DPWG76761 (WG76392) and were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification. Two results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW14A-GW-316-326-01-041621 | L34828-1 | 13C2-4:2 FTS | 255 | No Qualifiers |
| MW14A-GW-316-326-01-041621 | L34828-1 | 13C2-6:2 FTS | 201 | No Qualifiers |
| MW14B-GW-60-70-01-041621 | L34828-2 | 13C2-4:2 FTS | 225 | No Qualifiers |
| MW4A-GW-140-160-01-041521 | L34828-3 | 13C2-4:2 FTS | 205 | No Qualifiers |
| MW4A-GW-140-160-02-041521 | L34828-4 | 13C2-4:2 FTS | 208 | No Qualifiers |
| MW1A-GW-360-370-01-041621 | L34828-5 | 13C2-4:2 FTS | 379 | No Qualifiers |
| MW1A-GW-360-370-01-041621 | L34828-5 | 13C2-6:2 FTS | 305 | No Qualifiers |
| MW1C-GW-51-55-01-042121 | L34828-6 | 13C2-4:2 FTS | 215 | No Qualifiers |
| MW2A-GW-240-263-01-041921 | L34828-8 | 13C2-4:2 FTS | 240 | No Qualifiers |
| MW2A-GW-240-263-01-041921 | L34828-8 | 13C2-6:2 FTS | 203 | No Qualifiers |
| MW2A-GW-240-263-01-041921 | L34828-8 | d9-NEt-FOSE | 46.1 | UJ |
| MW17A-GW-230-240-01-041921 | L34828-9 | 13C2-PFTeDA | 42.4 | UJ |
| MW17A-GW-230-240-01-041921 | L34828-9 | 13C2-4:2 FTS | 451 | No Qualifiers |
| MW17A-GW-230-240-01-041921 | L34828-9 | 13C2-6:2 FTS | 202 | No Qualifiers |
| MW17A-GW-230-240-01-041921 | L34828-9 | D5-N-EtFOSA | 44.3 | No Qualifiers |
| MW17A-GW-230-240-01-041921 | L34828-9 | d7-NMe-FOSE | 36.6 | No Qualifiers |
| MW17A-GW-230-240-01-041921 | L34828-9 | d9-NEt-FOSE | 31.4 | No Qualifiers |
| MW17B-GW-90-100-01-041921 | L34828-10 | 13C2-PFTeDA | 41.1 | UJ |
| MW17B-GW-90-100-01-041921 | L34828-10 | 13C2-4:2 FTS | 216 | No Qualifiers |
| MW17B-GW-90-100-01-041921 | L34828-10 | D3-N-MeFOSA | 39.0 | UJ |
| MW17B-GW-90-100-01-041921 | L34828-10 | D5-N-EtFOSA | 34.2 | UJ |
| MW17B-GW-90-100-01-041921 | L34828-10 | d7-NMe-FOSE | 38.9 | UJ |
| MW17B-GW-90-100-01-041921 | L34828-10 | d9-NEt-FOSE | 29.8 | UJ |
| MW3B-GW-110-130-01-041921 | L34828-11 | 13C2-4:2 FTS | 247 | No Qualifiers |
| MW5B-GW-110-120-01-041921 | L34828-12 | 13C2-4:2 FTS | 252 | No Qualifiers |
| MW5B-GW-110-120-01-041921 | L34828-12 | d9-NEt-FOSE | 49.4 | UJ |
| MW5A-GW-210-220-01-041921 | L34828-13 | 13C4-PFBA | 3.96 | R |

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW5A-GW-210-220-01-041921 | L34828-13 | 13C5-PFPeA | 47.8 | J+ |
| MW5A-GW-210-220-01-041921 | L34828-13 | 13C2-4:2 FTS | 226 | No Qualifiers |
| MW6A-GW-185-192-01-042121 | L34828-14 | 13C4-PFBA | 14.1 | R |
| MW6A-GW-185-192-01-042121 | L34828-14 | 13C2-4:2 FTS | 254 | No Qualifiers |
| MW6A-GW-185-192-01-042121 | L34828-14 | d9-NEt-FOSE | 45.7 | UJ |
| MW6B-GW-140-150-01-041921 | L34828-15 | 13C2-4:2 FTS | 227 | No Qualifiers |
| MW3A-GW-230-250-01-041921 | L34828-16 | 13C2-4:2 FTS | 224 | No Qualifiers |
| MW9B-GW-90-100-01-042321 | L34828-17 | 13C2-4:2 FTS | 243 | No Qualifiers |
| MW15B-GW-215-225-01-041921 | L34828-18 | 13C2-4:2 FTS | 407 | No Qualifiers |
| MW15B-GW-215-225-01-041921 | L34828-18 | 13C2-6:2 FTS | 205 | No Qualifiers |
| MW15A-GW-330-340-01-041921 | L34828-19 | 13C2-4:2 FTS | 245 | No Qualifiers |
| MW7A-GW-200-210-01-041921 | L34828-20 | 13C4-PFBA | 20.6 | J+ |
| MW7A-GW-200-210-01-041921 | L34828-20 | 13C2-4:2 FTS | 237 | No Qualifiers |
| MW7A-GW-200-210-01-041921 | L34828-20 | d9-NEt-FOSE | 48.3 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was not associated with this data set. Results for an LCSD was provided to assess laboratory precision.

**LCS/LCSD Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits except as listed in the table below.

| LCS/LCSD IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG76457-102 | NFDHA | **55.4**/71.9 | 70-130 | Associated results were nondetect and were qualified UJ. |

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Sample MW4A-GW-140-160-02-041521 (L34828-4) was collected as a field duplicate of MW4A-GW-140-160-01-041521 (L34828-3). RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the reporting limit (RL).

**Quantitation and Result Verifications**

The PFOS result for sample MW14B-GW-60-70-01-041621 (L34828-2) exceeded the instrument calibration range and was reported from a five times dilution.

The PFOS result for sample MW15B-GW-215-225-01-041921 (L34828-18) was reported from re-analysis since carry-over was present during the initial analysis.

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| MW1A-GW-360-370-01-041621<br>MW17A-GW-230-240-01-041921<br>MW6A-GW-185-192-01-042121 | L34828-5<br>L34828-9<br>L34828-14 | PFOS | Yes | The ion ratio did not meet criteria, result qualified estimated biased high (J+) for one (or more) isomers. |
| MW2A-GW-240-263-01-041921 | L34828-8 | PFOS | Yes | The signal to noise ratio did not meet criteria for two of the branched isomers, and one of these branch isomer also did not meet ion ratio criteria. The result was qualified estimated biased high (J+), |
| MW17B-GW-90-100-01-041921<br>MW5A-GW-210-220-01-041921<br>MW3A-GW-230-250-01-041921 | L34828-10<br>L34828-13<br>L34828-16 | PFOS | Yes | Confirmation ion not present for one of the branched ions. |
| MW6B-GW-140-150-01-041921<br>MW15A-GW-330-340-01-041921<br>MW7A-GW-200-210-01-041921 | L34828-15<br>L34828-19<br>L34828-20 | PFOS | Yes | Confirmation ion not present for one of the branched ions. One of the isomer did not meet ion ratio criteria. |
| MW15B-GW-215-225-01-041921 | L34828-18 i | PFOS | Yes | Confirmation ion not present for one of the branched ions. One of the isomer did not meet ion ratio criteria. Signal to noise criteria not met. |
| MW1C-GW-51-55-01-042121 | L34828-6 | PFOS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak (one or more) that did not meet the ion ratio criteria. |
| MW14A-GW-316-326-01-041621<br>MW14B-GW-60-70-01-041621<br>MW1C-GW-51-55-01-042121<br>MW1C-GW-40-45-01-042221<br>MW3B-GW-110-130-01-041921 | L34828-1<br>L34828-2<br>L34828-6<br>L34828-7<br>L34828-11 | PFNA | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak that did not meet the signal to noise criteria. |
| MW1A-GW-360-370-01-041621<br>MW6B-GW-140-150-01-041921 | L34828-5<br>L34828-15 | PFOA | No | |
| MW2A-GW-240-263-01-041921<br>MW5A-GW-210-220-01-041921<br>MW6B-GW-140-150-01-041921 | L34828-8<br>L34828-13<br>L34828-15 | PFHxS | No | |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All Samples | All PFAS Analytes | ng/L | Detects: J-<br>Nondetect: UJ | sr |
| MW17A-GW-230-240-01-041921 (L34828-9) | PFTeDA | ng/L | UJ | s |
| MW2A-GW-240-263-01-041921 (L34828-8)<br>MW5B-GW-110-120-01-041921 (L34828-12)<br>MW6A-GW-185-192-01-042121 (L34828-14)<br>MW7A-GW-200-210-01-041921 (L34828-20) | NEt-FOSE | ng/L | UJ | s |
| MW17B-GW-90-100-01-041921 (L34828-10) | PFTeDA<br>N-MeFOSA<br>N-EtFOSA<br>NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| MW5A-GW-210-220-01-041921 (L34828-13)<br>MW6A-GW-185-192-01-042121 (L34828-14) | PFBA | ng/L | R | s |
| MW7A-GW-200-210-01-041921 (L34828-20) | PFBA | ng/L | J+ | s |
| MW5A-GW-210-220-01-041921 (L34828-13) | PFPeA | ng/L | J+ | s |
| All samples | NFDHA | ng/L | UJ | c,l |
| MW1A-GW-360-370-01-041621 (L34828-5)<br>MW2A-GW-240-263-01-041921 (L34828-8)<br>MW17A-GW-230-240-01-041921 (L34828-9)<br>MW17B-GW-90-100-01-041921 (L34828-10)<br>MW5A-GW-210-220-01-041921 (L34828-13)<br>MW6A-GW-185-192-01-042121 (L34828-14)<br>MW6B-GW-140-150-01-041921 (L34828-15)<br>MW15B-GW-215-225-01-041921 (L34828-18)<br>MW15A-GW-330-340-01-041921 (L34828-19)<br>MW7A-GW-200-210-01-041921 (L34828-20) | PFOS | ng/L | J+ | em |
| MW1C-GW-51-55-01-042121 (L34828-6) | PFOS | ng/L | J+ | q |
| MW1A-GW-360-370-01-041621 (L34828-5)<br>MW6B-GW-140-150-01-041921 (L34828-15) | PFOA | ng/L | J+ | q |
| MW14A-GW-316-326-01-041621 (L34828-1)<br>MW14B-GW-60-70-01-041621 (L34828-2)<br>MW1C-GW-51-55-01-042121 (L34828-6)<br>MW1C-GW-40-45-01-042221 (L34828-7)<br>MW3B-GW-110-130-01-041921 (L34828-11) | PFNA | ng/L | J+ | q |
| MW4A-GW-140-160-01-041521 (L34828-3)<br>MW5A-GW-210-220-01-041921 (L34828-13)<br>MW6B-GW-140-150-01-041921 (L34828-15) | PFHxS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

- J Estimated (+/- indicates high or low bias)
- R Rejected
- UJ Estimated reporting limit

(2): Reason Codes:

- c calibration
- em Estimated maximum concentration
- l Laboratory control sample

| | |
|---|---|
| q | Quantitation |
| sr | Sample receipt |
| s | Surrogate (labeled compound) |

# Attachment A

**SGS AXYS DPWG76949** 9/26/21 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | | | |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW3B-GW-110-130-02-041921 | PFOS | 910338 | 20130 | 1 | 704.11 | 705 | 0.517 | 0.004 | L34879-4 |
| MW14D-GW-6-21-01-050321 | PFOS | 632238 | 12271 | 1 | 802.20 | 803 | 0.517 | 0.004 | L34879-10 |
| LCS (WG76499-102) | PFOS | 14108 | 23471 | 1 | 4.84 | 4.24 | 1.00 | 0.004 | WG76499-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/5/21 9:20 | 16551 | 27987 | 4.760 | 4.76 | 5.00 | 95.21 | 95.2% |
| CCV 6/5/21 13:16 | 16120 | 28239 | 4.595 | 4.60 | 5.00 | 91.90 | 91.9% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG76499-101): | PFOS | 0.031 | <0.4 | verified <0.4 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG76949 (Analysis WG76499) dated 6/15/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 9/29/2021 |

The samples listed below were collected at the Project 1007 Site on April 19 through May 3, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW1B-GW-100-120-01-042721 | | 4/27/2021 | L34879-2 |
| MW2E-GW-90-100-01-042721 | | 4/27/2021 | L34879-3 |
| MW3B-GW-110-130-02-041921 | Field Duplicate of MW3B-GW-110-130-01-041921 | 4/19/2021 | L34879-4 |
| MW9A-GW-140-150-01-042921 | | 4/29/2021 | L34879-5 |
| MW9A-GW-140-150-02-042921 | Field Duplicate of MW9A-GW-140-150-01-042921 | 4/29/2021 | L34879-6 |
| MW10C-GW-10-20-01-050321 | | 5/3/2021 | L34879-7 |
| MW12A-GW-350-360-01-042121 | | 4/21/2021 | L34879-8 |
| MW14C-GW-16-36-01-042321 | | 4/23/2021 | L34879-9 |
| MW14D-GW-6-21-01-050321 | | 5/3/2021 | L34879-10 |
| MW17C-GW-38.5-48.5-01-050321 | | 5/3/2021 | L34879-11 |
| **Trip Blank:** | | | |
| 786743853090 | Trip Blank | 5/3/2021 | L34879-1 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds (Surrogates) |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control sample (LCS) |
| ✗ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol ( ✗ ) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to labeled compounds recoveries, field imprecision and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

The trip blank field ID was updated to comply with MPCA chain-of-custody (CoC) requirements.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. ICALs were also evaluated utilizing the MPCA criteria of 75% to 125% recovery of the true value (except for the lowest calibration point which may be 70% to 130% recovery), and the recoveries were within the MPCA limits for calibration points used for quantitation, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| ICAL (2/22/21) | 6:2 FTS High (3600) Cal Std | 58.1% | 75-125% | Results were nondetect, and were not reported from the highest (and second highest) calibration point. These calibration standards were not used in the ICAL. No qualifiers. |
| | 8:2 FTS High (4000) Cal Std | 54.2% | 75-125% | |
| | N-MeFOSA High (2500) Cal Std | 68.3% | 75-125% | |
| | 3:3 FTCA High (4000) Cal Std | 174.3% | 75-125% | |
| | 5:3 FTCA 7:3 FTCA NFDHA High Cal Stds | NQ | 75-125% | |
| | PFTrDA High (1000) Cal Std | 48.8% | 75-125% | |
| | PFTeDA High (1000) Cal Std | 64.4% | 75-125% | |
| | PFTrDA 250 Cal Std | 72.8% | 75-125% | |
| | 13C3-PFBA High Cal Std | 49.4% | 50-200% | PFBA values were not quantitated using the highest standard. This analyte is a recovery standard (reported in instrument printouts only). No qualifiers. |

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. Results associated high recoveries were nondetects and were acceptable without qualification.

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW1B-GW-100-120-01-042721 | L34879-2 | 13C2-4:2 FTS | 258 | No Qualifiers |
| MW2E-GW-90-100-01-042721 | L34879-3 | 13C2-4:2 FTS | 264 | No Qualifiers |
| MW9A-GW-140-150-01-042921 | L34879-5 | 13C4-PFBA | 39.6 | J+ |
| MW9A-GW-140-150-01-042921 | L34879-5 | 13C2-4:2 FTS | 270 | No Qualifiers |
| MW9A-GW-140-150-02-042921 | L34879-6 | 13C2-4:2 FTS | 375 | No Qualifiers |

| Sample ID | Lab ID | Labeled Compound | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW9A-GW-140-150-02-042921 | L34879-6 | 13C2-6:2 FTS | 210 | No Qualifiers |
| MW9A-GW-140-150-02-042921 | L34879-6 | d9-NEt-FOSE | 44.4 | UJ |
| MW12A-GW-350-360-01-042121 | L34879-8 | 13C4-PFBA | 29.6 | UJ |
| MW12A-GW-350-360-01-042121 | L34879-8 | 13C2-4:2 FTS | 318 | No Qualifiers |
| MW14D-GW-6-21-01-050321 | L34879-10 | 13C2-PFTeDA | 48.7 | UJ |
| MW14D-GW-6-21-01-050321 | L34879-10 | D5-N-EtFOSA | 47.2 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was not associated with this data set. Results for an LCSD was provided to assess laboratory precision.

**LCS/LCSD Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples MW3B-GW-110-130-02-041921 and MW9A-GW-140-150-02-042921 were collected as duplicates of samples MW3B-GW-110-130-01-041921 and MW9A-GW-140-150-01-042921, respectively. The parent sample MW3B-GW-110-130-01-041921 (L34828-11) was reported in SDG DPWG76830 (WG76457). RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± the reporting limit (RL), with the exception of those indicated in bold in the table below. Results associated with field imprecision were qualified as estimated (J).

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **MW9A-GW-140-150-01-042921 / MW9A-GW-140-150-02-042921:** | | | |
| PFOS | 101 | 63.6 | **45.4** |

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| MW10C-GW-10-20-01-050321<br>MW12A-GW-350-360-01-042121 | L34879-7<br>L34879-8 | PFOS | Yes | The ion ratio did not meet criteria, result qualified estimated biased high (J+) for one (or more) isomers. |
| MW2E-GW-90-100-01-042721 | L34879-3 | PFDA | Yes | The ion ratio did not meet criteria, result qualified estimated biased high (J+). |
| MW14D-GW-6-21-01-050321 | L34879-10 | EtFOSAA | No | Confirmation ion not present for one of the branched ions. One of the isomer did not meet ion ratio criteria. Signal to noise criteria not met for all isomer. |
| MW9A-GW-140-150-01-042921<br>MW14C-GW-16-36-01-042321<br>MW14D-GW-6-21-01-050321<br>MW17C-GW-38.5-48.5-01-050321 | L34879-5<br>L34879-9<br>L34879-10<br>L34879-11 | PFOS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak (one or more) that did not meet the ion ratio criteria. |
| MW3B-GW-110-130-02-041921<br>MW14C-GW-16-36-01-042321<br>MW14D-GW-6-21-01-050321<br>MW17C-GW-38.5-48.5-01-050321 | L34879-4<br>L34879-9<br>L34879-10<br>L34879-11 | PFNA | No | The result was qualified as estimated biased high (J+) as it included quantitation of a peak that did not meet the signal to noise criteria. |
| MW9A-GW-140-150-01-042921 | L34879-5 | PFOA | No | |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW9A-GW-140-150-01-042921 (L34879-5)<br>MW12A-GW-350-360-01-042121 (L34879-8) | PFBA | ng/L | J+<br>UJ | s |
| MW9A-GW-140-150-02-042921 (L34879-6) | NEt-FOSE | ng/L | UJ | s |
| MW14D-GW-6-21-01-050321 (L34879-10) | PFTeDA<br>N-EtFOSA | ng/L | UJ | s |
| MW10C-GW-10-20-01-050321 (L34879-7)<br>MW12A-GW-350-360-01-042121 (L34879-8) | PFOS | ng/L | J+ | em |
| MW2E-GW-90-100-01-042721 (L34879-3) | PFDA | ng/L | J+ | em |
| MW9A-GW-140-150-01-042921 (L34879-5) | PFOA | ng/L | J+ | q |
| MW3B-GW-110-130-02-041921 (L34879-4)<br>MW14C-GW-16-36-01-042321 (L34879-9)<br>MW14D-GW-6-21-01-050321 (L34879-10)<br>MW17C-GW-38.5-48.5-01-050321 (L34879-11) | PFNA | ng/L | J+ | q |
| MW9A-GW-140-150-01-042921 (L34879-5)<br>MW14C-GW-16-36-01-042321 (L34879-9)<br>MW14D-GW-6-21-01-050321 (L34879-10)<br>MW17C-GW-38.5-48.5-01-050321 (L34879-11) | PFOS | ng/L | J+ | q |
| MW14D-GW-6-21-01-050321 (L34879-10) | EtFOSAA | ng/L | J+ | q |
| MW9A-GW-140-150-01-042921 (L34879-5)<br>MW9A-GW-140-150-02-042921 (L34879-6) | PFOS | ng/L | J | fd |

(1): Data Validation Qualifiers:

| | |
|---|---|
| J | Estimated (+/- indicates high or low bias) |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| em | Estimated maximum concentration |
| fd | Field duplicate |
| q | Quantitation |
| s | Surrogate (labeled compound) |

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77059 (WG76810)**

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at a temperature greater than 6 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| FW09-SED-12-24-01-033121 | L34671-1 | 15 |
| FW10-SED-12-24-01-033121 | L34671-4 | 15 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/12/21 2:25 | N-EtFOSE | 162% | 70-130% | Associated results were nondetects were acceptable without qualification. |
| | ADONA | 133% | 70-130% | |
| | 9Cl-PF3ONS | 131% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PW05-SED-12-24-01-031521 | L34606-15 | D3-N-MeFOSA | 40.6 | J+ |
| PW05-SED-12-24-01-031521 | L34606-15 | D5-N-EtFOSA | 28.7 | J+ |
| FW10-SED-12-24-01-033121 | L34671-4 | D5-N-EtFOSA | 46.2 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **PW01-SED-12-24-01-031521/PW01-SED-12-24-02-031521:** | | | |
| N-EtFOSAA | 0.266 | 0.065 J | >±RL |
| PFBA | 4.74 | 2.37 | 66.7 |
| PFDoA | 0.234 | 0.074 J | >±RL |
| PFDS | 0.897 | 0.307 | 98 |
| PFHpA | 0.506 | 0.284 | 56.2 |
| PFHpS | 0.584 | 0.333 | 54.7 |
| PFHxS | 0.451 | 0.218 | 69.7 |
| PFOA | 8.3 | 3.02 | 93.3 |

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| PFOSA | 2.22 | 1.12 | 65.9 |
| PFPeA | 0.547 | 0.297 J | >±RL |
| PFUnA | 0.299 | 0.117 J | >±RL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PW01-SED-12-24-01-031521 | L34606-1 | MeFOSAA |
| PW01-SED-12-24-02-031521 | L34606-2 | PFDoS |
| PW03-SED-12-24-01-031521 | L34606-9 | PFDoA |
| PW04-SED-12-24-01-031521 | L34606-12 | PFPeS |
| PW05-SED-12-24-01-031521 | L34606-15 | N-EtFOSA |
| PW05-SED-24-48-01-031521 | L34606-16 | MeFOSAA |
| FW09-SED-12-24-01-033121 | L34671-1 | PFDS |
| FW10-SED-12-24-01-033121 | L34671-4 | PFDoS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77059 Report date (mm/dd/yyyy): 6/23/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/1/2021

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | Two of the samples were received at an elevated temperature of 15 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | A QC sample (MS/MSD) was submitted with this SDG; however, was not taken off of hold. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150% |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD ok<br>FDs: PW01-SED-12-24-01-031521/PW01-SED-12-24-02-031521<br>PW10-SED-12-24-01-031521/PW10-SED-12-24-02-031521 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>See data qualifications summary for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/1/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77060 (WG76811)**

**Chain of custody preservation, and holding time (Section 1C):** The samples listed below were received at temperatures greater than 6 °C. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

| Sample ID | Lab ID | Sample Receipt Temperature (°C) |
|---|---|---|
| FW10-SED-24-36-01-033121 | L34671-6 | 15 |
| FW10-SED-36-48-01-033121 | L34671-8 | 15 |
| FW11-SED-12-24-01-033121 | L34671-10 | 15 |
| FW11-SED-24-48-01-033121 | L34671-11 | 15 |
| FW06-SED-12-24-01-040621 | L34748-4 | 7.4 |
| FW07-SED-24-36-01-040621 | L34748-5 | 7.4 |
| FW07-SED-24-36-02-040621 | L34748-6 | 7.4 |
| FW07-SED-36-48-01-040621 | L34748-7 | 7.4 |
| FW07-SED-36-48-02-040621 | L34748-8 | 7.4 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/12/21 2:25 | N-EtFOSE | 162% | 70-130% | Associated results were nondetects were acceptable without qualification. |
| | ADONA | 133% | 70-130% | |
| | 9Cl-PF3ONS | 131% | 70-130% | |
| CCV 6/15/21 19:51 | NFDHA | 69.2% | 70-130% | NFDHA was not reported from this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW11-SED-12-24-01-033121 | L34671-10 | D5-N-EtFOSA | 47.0 | UJ |
| FW11-SED-24-48-01-033121 | L34671-11 | D3-N-MeFOSA | 48.3 | UJ |
| FW11-SED-24-48-01-033121 | L34671-11 | D5-N-EtFOSA | 42.2 | UJ |
| FW06-SED-12-24-01-040621 | L34748-4 | D3-N-MeFOSA | 46.3 | J+ |
| FW06-SED-12-24-01-040621 | L34748-4 | D5-N-EtFOSA | 36.0 | J+ |
| FW07-SED-24-36-01-040621 | L34748-5 | D3-N-MeFOSA | 48.5 | J+ |
| FW07-SED-24-36-01-040621 | L34748-5 | D5-N-EtFOSA | 37.4 | J+ |
| FW07-SED-24-36-02-040621 | L34748-6 | D3-N-MeFOSA | 49.5 | J+ |
| FW07-SED-24-36-02-040621 | L34748-6 | D5-N-EtFOSA | 37.8 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| FW03-SED-12-24-01-041421 | L34799-4 K | D3-N-MeFOSA | 40.7 | J+ |
| FW03-SED-12-24-01-041421 | L34799-4 K | D5-N-EtFOSA | 31.9 | UJ |
| FW04-SED-24-36-01-041421 | L34799-7 | D3-N-MeFOSA | 42.1 | J+ |
| FW04-SED-24-36-01-041421 | L34799-7 | D5-N-EtFOSA | 31.8 | UJ |
| FW05-SED-24-36-01-041421 | L34799-10 | D5-N-EtFOSA | 47.5 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| FW11-SED-12-24-01-033121 | L34671-10 | N-MeFOSA |
| FW11-SED-12-24-01-033121 | L34671-10 | PFDoS |
| FW06-SED-12-24-01-040621 | L34748-4 | N-EtFOSA |
| FW07-SED-24-36-01-040621 | L34748-5 | N-EtFOSA |
| FW07-SED-24-36-01-040621 | L34748-5 | PFDoS |
| FW07-SED-24-36-02-040621 | L34748-6 | N-EtFOSA |
| FW07-SED-24-36-02-040621 | L34748-6 | PFDoS |
| FW07-SED-36-48-01-040621 | L34748-7 | N-MeFOSAA |
| FW07-SED-36-48-02-040621 | L34748-8 | PFDoS |
| FW07-SED-36-48-01-040621 | L34799-7 | N-MeFOSA |
| FW05-SED-12-24-01-041421 | L34799-9 | N-MeFOSA |

For sample FW03-SED-12-24-01-041421 (L34799-4 K). the labeled analogs were not added prior to extraction (inadvertently missed) and were added after extraction, but before analysis. Results for this sample were qualified as estimated (UJ/J).

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist


Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560


*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77060 Report date (mm/dd/yyyy): 6/23/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/9/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Laboratory indicated the container label and COC did not match for lab ID L34748-8. The sample was logged in using the ID on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | Nine of the samples were received at an elevated temperatures of 7.4 °C and 15 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | QC samples (MS/MSDs) were submitted with this SDG; however, were not taken off of hold. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances. No qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | MPCA PFAS other limits used (matrix listed as solid): 50% to 150% |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD ok<br>FDs: FW07-SED-24-36-01-040621/FW07-SED-24-36-02-040621<br>FW07-SED-36-48-01-040621/FW07-SED-36-48-02-040621 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/9/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77811 (WG77446)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 9.8 °C, 9.9 °C, and 10 °C; and were above the 6 °C criteria. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/17/21 16:40 | NFDHA | 65.3% | 70-130% | NFDHA was not reported from this CCV. |
| CCV 8/19/21 8:16 | NFDHA | 55.2% | 70-130% | NFDHA was not reported from this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC5-WAT-BULK-01-071321 | L35278-26 | D3-MeFOSAA | 42.7 | J+ |
| RC5-WAT-BULK-02-071321 | L35278-27 | D3-MeFOSAA | 41.7 | J+ |
| RC5-WAT-BULK-03-071321 | L35278-28 | D3-MeFOSAA | 44.1 | J+ |
| RC14-WAT-BULK-01-071321 | L35278-29 | 13C4-PFBA | 20.0 | J+ |
| RC21-WAT-BULK-01-071421 | L35278-30 | 13C4-PFBA | 43.2 | J+ |
| RC23-WAT-BULK-01-071321 | L35278-31 | 13C2-PFTeDA | 46.2 | UJ |
| RC23-WAT-BULK-01-071321 | L35278-31 | D3-MeFOSAA | 32.1 | UJ |
| EP17C-WAT-BULK-01-071321 | L35278-32 | 13C4-PFBA | 2.74 | R |
| EP17C-WAT-BULK-01-071321 | L35278-32 | 13C5-PFPeA | 45.2 | J+ |
| EP17C-WAT-BULK-01-071321 | L35278-32 | D3-MeFOSAA | 47.4 | UJ |
| PZDS-GW-20-30-01-071521 | L35278-35 | D3-N-MeFOSA | 43.9 | UJ |
| PZDS-GW-20-30-01-071521 | L35278-35 | D5-N-EtFOSA | 43.2 | UJ |
| PZDS-GW-20-30-02-071521 | L35278-36 | D5-N-EtFOSA | 47.8 | UJ |
| PZED-GW-16-21-01-071521 | L35278-39 | D3-MeFOSAA | 44.9 | UJ |
| PZJS-GW-15-25-01-071521 | L35278-40 | D5-N-EtFOSA | 48.3 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-071321:** | | | | | |
| PFBA | **57.4/60.9** | 70-130 | 0.11 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFHxA | 70.3/**279** | 70-130 | 12.1 | 30 | |
| PFHpA | **17.7/--** | 70-130 | 19.5 | 30 | |
| PFOA | **187/--** | 70-130 | 7.60 | 30 | |
| PFHxS | **148/145** | 70-130 | 0.313 | 30 | |
| PFOS | **1130/500** | 70-130 | 3.19 | 30 | |
| PFBS | **246**/79.2 | 70-130 | **31.1** | 30 | The detects for samples RC5-WAT-BULK-01-071321, RC5-WAT-BULK-02-071321 and RC5-WAT-BULK-03-071321 were qualified as estimated biased high (J+). |
| PFHpS | **163**/108 | 70-130 | 12.9 | 30 | |
| PFPeS | 124/**134** | 70-130 | 2.44 | 30 | |
| EtFOSAA | 98.7/**150** | 70-130 | 16.4 | 30 | |
| NFDHA | **43.5/58.7** | 70-130 | **30.4** | 30 | The results for samples RC5-WAT-BULK-01-071321, RC5-WAT-BULK-02-071321 and RC5-WAT-BULK-03-071321 were nondetect and qualified UJ. |
| 11Cl-PF3OUdS | 94.6/**65.0** | 70-130 | **36.3** | 30 | |
| 9Cl-PF3ONS | 110/77.2 | 70-130 | **34.3** | 30 | The associated results were nondetect and were acceptable without qualification. |
| ADONA | 120/70.6 | 70-130 | **33.7** | 30 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **PZDS-GW-20-30-01-071521 / PZDS-GW-20-30-02-071521:** | | | |
| 6:2 FTS | 7.15 | 1.35 U | >±RL |
| PFOSA | 2.13 | 1.55 | 31.5 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC5-WAT-BULK-01-071321 | L35278-26 | PFNS |
| RC5-WAT-BULK-02-071321 | L35278-27 | PFNS |
| RC14-WAT-BULK-01-071321 | L35278-29 i | PFPeS |
| RC21-WAT-BULK-01-071421 | L35278-30 | PFPeS |

The case narrative indicates that samples PZCS-GW-6-11-01-071421, RC14-WAT-BULK-01-071321 and EP17C-WAT-BULK-01-071321 were reanalyzed due to carry-over concerns. Some of the results required reporting from the re-analysis.

The PFOS result for sample RC23-WAT-BULK-01-071321 was reported from a five times dilution analysis due to a high sample concentration.

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77811 Report date (mm/dd/yyyy): 8/27/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/27/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at elevated temperatures of 9.8 °C, 9.9 °C, and 10 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances. No qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Trip blanks (L35278-1, L35278-2, and L35278-3) were listed on the COCs, and reported in WG77448. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-071321 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | No rerun. MS/MSD exceedance discussed in case narrative. |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | No rerun. MS/MSD exceedance discussed in case narrative. |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RC5-WAT-BULK-03-071321.<br>FDs: RC5-WAT-BULK-01-071321/RC5-WAT-BULK-02-071321/RC5-WAT-BULK-03-07132<br>PZDS-GW-20-30-01-071521/PZDS-GW-20-30-02-071521 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☐ | ☒ | ☐ | MPCA limit: 30% RPD for waters<br>See data qualifications summary for |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

**One result was rejected (R) due to a severely low recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/27/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77917 (WG77477)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 9.8 °C, 9.9 °C, and 10 °C; and were above the 6 °C criteria. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/15/21 3:49 | NFDHA | 58.0% | 70-130% | Associated results were nondetect and qualified UJ. |
| CCV 8/15/21 7:31 | HFPO-DA | 64.7% | 70-130% | Associated results were nondetect and qualified UJ. |
| CCV 8/17/21 16:40 | NFDHA | 66.9% | 70-130% | NFDHA was not reported from this CCV. |
| CCV 8/17/21 19:03 | HFPO-DA | 67.2% | 70-130% | HFPO-DA was not reported from this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW1A-GW-360-370-01-071621 | L35278-42 | 13C4-PFBA | 22.8 | J+ |
| MW2E-GW-90-100-01-071621 | L35278-44 | 13C4-PFBA | 21.4 | J+ |
| MW3A-GW-230-250-01-071621 | L35278-45 | 13C4-PFBA | 19.2 | R |
| RC3N-WAT-BULK-01-071521 | L35278-54 | 13C4-PFBA | 39.3 | J+ |
| RC3CUL-WAT-BULK-01-071521 | L35278-55 | 13C4-PFBA | 47.6 | J+ |
| RC3MAN-WAT-BULK-01-071521 | L35278-56 | 13C4-PFBA | 49.6 | J+ |
| RC3MAN-WAT-BULK-01-071521 | L35278-56 | D3-MeFOSAA | 48.9 | UJ |
| RC5-WAT-BULK-01-071521 | L35278-57 | 13C4-PFBA | 39.0 | J+ |
| RC5-WAT-BULK-01-071521 | L35278-57 | D3-MeFOSAA | 37.7 | UJ |
| EP16-WAT-BULK-01-071621 | L35278-58 | 13C4-PFBA | 32.6 | J+ |
| EP24-WAT-BULK-01-071521 | L35278-60 | 13C2-PFTeDA | 48.8 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **PZGD-GW-35-40-03-071321:** | | | | | |
| PFBA | **38.7/67.1** | 70-130 | 7.84 | 30 | The detects for samples PZGD-GW-35-40-01-071321, PZGD-GW-35-40-02-071321, and PZGD-GW-35-40-03-071321 were qualified as estimated biased low (J-). |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **PZGD-GW-35-40-01-071321 / PZGD-GW-35-40-02-071321 / PZGD-GW-35-40-03-071321:** | | | | |
| PFHxA | 2.97 | 4.01 | 3.04 | 31.1 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW1A-GW-360-370-01-071621 | L35278-42 | PFDS |
| MW1A-GW-360-370-01-071621 | L35278-42 | PFOS |
| MW1B-GW-100-120-01-071621 | L35278-43 | PFOS |
| PZHD-GW-21-26-01-071321 | L35278-53 | PFNA |

The case narrative indicates that samples MW2E-GW-90-100-01-071621 and EP16-WAT-BULK-01-071621 were reanalyzed due to carry-over concerns. Some of the results required reporting from the re-analysis.

The PFOS result for sample RC5-WAT-BULK-01-071521 (L352478-57), was reported from a five times dilution analysis to bring the sample concentration within the instrument calibration range.

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77917 Report date (mm/dd/yyyy): 9/2/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/28/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at elevated temperatures of 9.5 °C, 10 °C, and 10.4 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Trip blanks (L35278-3, L35278-4, L35278-5) were listed on the COCs, and reported in WG77448. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZGD-GW-35-40-03-071321 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | No rerun. MS/MSD exceedance discussed in case narrative. |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | No rerun. MS/MSD exceedance discussed in case narrative. |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>FDs: RC5-WAT-BULK-01-071321/RC5-WAT-BULK-02-071321/RC5-WAT-BULK-03-07132<br>PZDS-GW-20-30-01-071521/PZDS-GW-20-30-02-071521 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | MPCA limit: 30% RPD for waters<br>See data qualifications summary for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

**One result was rejected (R) due to a severely low recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/28/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77923 (WG77554)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD3-SED-0-6-01-071921 | L35316-2 | D3-N-MeFOSA | 42.0 | J+ |
| OD3-SED-0-6-01-071921 | L35316-2 | D5-N-EtFOSA | 36.0 | UJ |
| OD6-SED-0-6-01-072021 | L35316-8 | D5-N-EtFOSA | 46.7 | UJ |
| OD7-SED-0-6-01-071921 | L35316-9 | D5-N-EtFOSA | 48.3 | UJ |
| OD9-SED-0-6-01-071921 | L35316-11 | D5-N-EtFOSA | 45.7 | UJ |
| OD10-SED-0-6-01-072221 | L35316-12 | D5-N-EtFOSA | 46.1 | UJ |
| OD11-SED-0-6-01-071921 | L35316-13 | D3-N-MeFOSA | 48.1 | UJ |
| OD11-SED-0-6-01-071921 | L35316-13 | D5-N-EtFOSA | 38.2 | UJ |
| OD11-SED-0-6-02-071921 | L35316-14 | D3-N-MeFOSA | 34.3 | UJ |
| OD11-SED-0-6-02-071921 | L35316-14 | D5-N-EtFOSA | 24.9 | UJ |
| OD12-SED-0-6-01-071921 | L35316-16 | D5-N-EtFOSA | 49.1 | UJ |
| TL1-SED-0-6-01-071921 | L35316-17 | D3-N-MeFOSA | 45.8 | UJ |
| TL1-SED-0-6-01-071921 | L35316-17 | D5-N-EtFOSA | 36.2 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OD3-SED-0-6-01-071921 | L35316-2 | N-MeFOSAA |
| OD5-SED-0-6-01-071921 | L35316-7 | N-EtFOSAA |
| OD6-SED-0-6-01-072021 | L35316-8 | PFHxA |
| OD8-SED-0-6-01-072021 | L35316-10 | N-MeFOSAA |
| OD9-SED-0-6-01-071921 | L35316-11 | PFHxA |
| OD10-SED-0-6-01-072221 | L35316-12 | N-EtFOSAA |
| OD11-SED-0-6-01-071921 | L35316-13 | N-MeFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77923 Report date (mm/dd/yyyy): 8/31/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/27/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Trip blank (L35316-1) was listed on the COCs. Results for this trip blank were not reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OD4-SED-0-6-03-072021 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>FDs: OD4-SED-0-6-01-072021/OD4-SED-0-6-02-072021/OD4-SED-0-6-03-072021<br>OD11-SED-0-6-01-071921/OD11-SED-0-6-02-071921 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Reduced sample size used for OD3-SED-0-6-01-071921 (L35316-2). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/28/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG77924 (WG77555)**

**Chain of custody preservation, and holding time (Section 1C):** The samples, except for OD11-SED-0-6-03-071921 (L35316-15 (A)), were received at a temperature of 10 °C and did not meet the criteria of ≤6 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/18/21 19:38 (Closing CCV) | NFDHA | 61.3% | 70-130% | All samples were associated with this CCV. Detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG77555-101 | PFOSA | 0.042 J | Associated detects within 10 times the method blank concentration were qualified B:<br><br>OD11-SED-0-6-03-071921 (L35316-15 (A))<br>TL2-SED-0-6-01-071921 (L35316-18)<br>CL1-SED-0-6-01-072021 (L35316-20)<br>CL2-SED-0-6-01-072021 (L35316-21)<br>CL2-SED-0-6-02-072021 (L35316-22) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD11-SED-0-6-03-071921 | L35316-15 (A) | D5-N-EtFOSA | 45.7 | UJ |
| TL2-SED-0-6-01-071921 | L35316-18 | 13C2-PFTeDA | 45.0 | UJ<br>PFTrDA: J+ |
| TL2-SED-0-6-01-071921 | L35316-18 | D3-N-MeFOSA | 34.9 | UJ |
| TL2-SED-0-6-01-071921 | L35316-18 | D5-N-EtFOSA | 28.0 | UJ |
| TL2-SED-0-6-01-071921 | L35316-18 | d9-NEt-FOSE | 47.1 | UJ |
| TL3-SED-0-6-01-072121 | L35316-19 | 13C2-PFTeDA | 49.3 | J+<br>PFTrDA: J+ |
| TL3-SED-0-6-01-072121 | L35316-19 | D3-N-MeFOSA | 41.1 | UJ |
| TL3-SED-0-6-01-072121 | L35316-19 | D5-N-EtFOSA | 31.1 | UJ |
| TL3-SED-0-6-01-072121 | L35316-19 | d9-NEt-FOSE | 42.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| GC1-SED-0-6-01-072221 | L35316-24 | D.3-N-MeFOSA | 43.5 | UJ |
| GC1-SED-0-6-01-072221 | L35316-24 | D5-N-EtFOSA | 30.7 | UJ |
| GC2A-SED-0-6-01-072221 | L35316-31 | D3-N-MeFOSA | 35.4 | UJ |
| GC2A-SED-0-6-01-072221 | L35316-31 | D5-N-EtFOSA | 27.7 | UJ |
| GC2A-SED-0-6-01-072221 | L35316-31 | d9-NEt-FOSE | 48.2 | UJ |
| BL3-SED-0-6-01-072121 | L35316-33 | D5-N-EtFOSA | 44.0 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **OD11-SED-0-6-03-071921:** | | | | | |
| 5:3 FTCA | 92.0/124 | 70-130 | **30.3** | 30 | The result for sample OD11-SED-0-6-03-071921 is nondetect, and is acceptable without qualification. |
| NFDHA | **57.1/69.1** | 70-130 | 19.9 | 30 | The result for sample OD11-SED-0-6-03-071921 is nondetect, and was qualified as estimated (UJ). |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OD11-SED-0-6-03-071921 | L35316-15 (A) | PFHxA |
| OD11-SED-0-6-03-071921 | L35316-15 (A) | MeFOSAA |
| TL3-SED-0-6-01-072121 | L35316-19 | PFHpA |
| TL3-SED-0-6-01-072121 | L35316-19 | PFPeS |
| CL1-SED-0-6-01-072021 | L35316-20 | PFHxA |
| CL2-SED-0-6-01-072021 | L35316-21 | MeFOSAA |
| GC1-SED-0-6-01-072221 | L35316-24 | EtFOSAA |
| GC4-SED-0-6-01-072121 | L35316-27 | MeFOSAA |
| GC5-SED-0-6-01-072121 | L35316-28 | MeFOSAA |
| BL1-SED-0-6-01-071921 | L35316-32 | EtFOSAA |

Results with both low and high bias were qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG77924 Report date (mm/dd/yyyy): 9/3/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/27/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples, except for OD11-SED-0-6-03-071921 (L35316-15), were received at a temperature of 10 °C and did not meet the criteria of ≤6 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | Trip blank (L35316-1) was listed on the COCs. Results for this trip blank were not reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.042 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OD11-SED-0-6-03-071921 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances and qualifications. |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs<br>FDs: CL2-SED-0-6-01-072021 / OD11-SED-0-6-02-071921 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>MSD - RPDs: See data qualifications summary for exceedances.<br>FDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/7/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG78554 (WG78095)**

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 10/1/21 18:49 | NFDHA | 55.0% | 70-130% | NFDHA results were nondetect and qualified UJ. |
| CCV 10/6/21 18:57 | NFDHA | 66.3% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ). Detects associated with high recoveries were qualified as estimated biased low (J-), as an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-090821 | L35587-1 i | D3-N-MeFOSA | 48.1 | UJ |
| TRIP-COOLER1-090821 | L35587-1 i | D5-N-EtFOSA | 48.3 | UJ |
| OD3-WAT-BULK-01-083021 | L35587-8 | 13C9-PFNA | 170 | J- |
| RC3-WAT-BULK-01-083021 | L35587-14 | D3-MeFOSAA | 48.5 | UJ |

**MS/MSD Exceedances (Section 6Av):** The MS/MSD associated with samples OD5-WAT-BULK-01-083021 (L35587-10) and OD5-WAT-BULK-02-083021 (L35587-11) was reported in 3000028832 (WG78096). MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **OD5-WAT-BULK-03-083021:** | | | | | |
| PFBA | **156 / 155** | 70-130 | 0.16 | 30 | The detects for samples OD5-WAT-BULK-01-083021 and OD5-WAT-BULK-02-083021 were qualified as estimated biased high (J+). |
| PFDoS | 72.3 **/ 62.3** | 70-130 | 13.3 | 30 | The results for samples OD5-WAT-BULK-01-083021 and OD5-WAT-BULK-02-083021 were nondetect and qualified UJ. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

The field triplicate (OD5-WAT-BULK-03-083021) was reported in DPWG78580 (WG78096). Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **OD5-WAT-BULK-01-083021 / OD5-WAT-BULK-02-083021 / OD5-WAT-BULK-03-083021** | | | | |

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| PFHxA | 11 | 7.08 | 8.42 | 44.4 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG78554 Report date (mm/dd/yyyy): 10/15/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 2/22/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at elevated temperatures of 6.2, 6.5, and 9.2 °C, and above the requirement of <6 °C. Results were not qualified, as the receipt temperatures were <10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-090821 (L35587-1 i)<br>TRIP-COOLER2-090821 (L35587-2)<br>TRIP-COOLER3-090821 (L35587-3)<br>TRIP-COOLER4-090821 (L35587-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | No detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method MLA-110 limits used: 50% to 150% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | An MS/MSD was desginated for sample OD5-WAT-BULK-03-083021 (L35587-12) which is reported in DPWG78580 (WG78096). |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - WAT-BULK-03-083021 (L35587-12) which is reported in DPWG78580 (WG78096).<br>FDs: OD5-WAT-BULK-01-083021 / OD5-WAT-BULK-02-083021 / OD5-WAT-BULK-03-083021 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☐ | ☐ | 30% RPD for waters<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using Method MLA-110MPCA limits and Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 2/22/2022

**SGS AXYS DPWG78555** 3/13/22 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | | | |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW9B-GW-90-100-01-083021 | PFOS | 172866 | 21582 | 1 | 117.01 | 117 | 0.551 | 0.004 | L35586-1 |
| MW9B-GW-90-100-02-083021 | PFOS | 185883 | 24155 | 1 | 113.87 | 114 | 0.544 | 0.004 | L35586-2 |
| LCS (WG76499-102) | PFOS | 16576 | 27756 | 1 | 4.81 | 4.81 | 1.00 | 0.004 | WG78095-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 10/1/21 16:12 | 17051 | 29501 | 4.653 | 4.65 | 5.00 | 93.05 | 93.1% |
| CCV 10/1/21 18:49 | 15412 | 27779 | 4.466 | 4.47 | 5.00 | 89.32 | 89.3% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG78095-101): | PFOS | 0.004 | <0.4 | verified <0.4 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG78555 (Analysis WG78095) dated 10/18/2021 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 3/13/2022 |

The samples listed below were collected at the Project 1007 Site on August 30 and September 1, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW9B-GW-90-100-01-083021 | | 8/30/2021 | L35586-1 |
| MW9B-GW-90-100-02-083021 | Field Duplicate | 8/30/2021 | L35586-2 |
| MW9B-GW-90-100-03-083021 | Field Triplicate; MS/MSD | 8/30/2021 | L35586-3 (A) |
| MW14C-GW-16-36-01-090121 | | 9/1/2021 | L35586-4 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance document listed above, and the analytical method (MLA-110).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✓ Sample Receipt and Holding times
✗ Initial calibration/continuing calibration verification
✓ Laboratory and trip blanks
✓ Labeled Compounds (Surrogates)
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓ Laboratory control sample (LCS)
✗ Field duplicates
✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to calibrations, matrix spikes, field imprecision and quantitations as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at SGS-AXYS intact, correctly preserved, and within the temperature criteria of ≤ 6 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for a number of analytes (and the two highest standards were excluded for NFDHA) from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

CCV recoveries were within 100 ± 30% of the true known value, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 10/1/21 18:49 | NFDHA | 55.0 | 70-130% | NFDHA results were nondetect and qualified UJ. |

A second source verification (SSV) was included in the raw instrument printouts; however, a summary form with the recoveries (and true value concentrations) for target compounds was not provided.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The associated trip blank (TRIP-COOLER4-090821, L35587-4) was reported in data package DPWG78554/ WG78095. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%.

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were reported for sample MW9B-GW-90-100-03-083021. MS/MSD recoveries and relative percent differences (RPDs) were within MPCA limits, except as listed in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW9B-GW-90-100-03-083021:** | | | | | |
| PFBA | **NR /** 98.0 | 70-130 | **31.9** | 30 | The detects for samples MW9B-GW-90-100-01-083021, MW9B-GW-90-100-02-083021, and MW9B-GW-90-100-03-083021 were qualified as estimated (J). |
| PFDoS | **68.8** / **69.7** | 70-130 | 1.01 | 30 | The results for samples MW9B-GW-90-100-01-083021, MW9B-GW-90-100-02-083021, and MW9B-GW-90-100-03-083021 were nondetect and qualified UJ. |
| NFDHA | **61.4** / **52.2** | 70-130 | 16.7 | 30 | |
| 9Cl-PF3ONS | **141** / 111 | 70-130 | 18.4 | 30 | The results for samples MW9B-GW-90-100-01-083021, MW9B-GW-90-100-02-083021, and MW9B-GW-90-100-03-083021 were nondetect, and were acceptable without qualification. |
| ADONA | **133** / 116 | 70-130 | 20.4 | 30 | |
| PFOA | 92.9 / **37.7** | 70-130 | 6.37 | 30 | The sample concentrations were greater than four times the spike concentration. No qualifiers. |
| PFOS | 106 / **137** | 70-130 | 2.23 | 30 | |

NR – not reported

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits of 70% to 130% recovery.

**Field Duplicate Results**

Field triplicate results were collected to assess the overall precision of field sampling and laboratory analysis. Samples MW9B-GW-90-100-01-083021, MW9B-GW-90-100-02-083021, and MW9B-GW-90-100-03-083021 were assess for field precision. RPDs were within the 30 percent limit for water samples, or were within ± the reporting limit (RL), with the exception of those indicated in bold in the table below. Results associated with field imprecision were qualified as estimated (J).

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **MW9B-GW-90-100-01-083021 / MW9B-GW-90-100-02-083021 / MW9B-GW-90-100-03-083021:** | | | | |
| PFBA | 260 | 125 | 328 | 85.4 |
| PFPeA | 6.3 | 3.09 | 7.94 | 84.0 |
| PFHxA | 5.15 | 2.57 | 5.33 | 63.4 |
| PFBS | 4.76 | 2.24 | 5.32 | 75.0 |
| PFPeS | 5.35 | 3.79 | 5.91 | 42.3 |

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. The review found that the sample concentrations reported in the raw data did not match the results reported for sample MW9B-GW-90-100-01-083021 (L35586-1), and audit change report documented the discrepancy. The audit change was required as an incorrect sample volume was used in the raw instrument printout (original quantitation).

The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| MW9B-GW-90-100-01-083021 | L35586-1 | PFOS | No | The PFOS result was qualified as estimated biased (J+) as it included quantitation of a branched isomer (PFOS-IV) that did not meet the ion ratio criteria. |
| MW14C-GW-16-36-01-090121 | L35586-4 | PFOS | No | The PFOS result was qualified as estimated biased (J+) as it included quantitation of a branched isomer (PFOS-IV) that did not meet the ion ratio criteria. |
| | | PFOSA | No | Signal to noise criteria not met for any of the isomers. |

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW14C-GW-16-36-01-090121 (L35586-4) | NFDHA | ng/L | UJ | c |
| MW9B-GW-90-100-01-083021 (L35586-1)<br>MW9B-GW-90-100-02-083021 (L35586-2)<br>MW9B-GW-90-100-03-083021 (L35586-3 (A)) | NFDHA | ng/L | UJ | c,ms |
| MW9B-GW-90-100-01-083021 (L35586-1)<br>MW9B-GW-90-100-02-083021 (L35586-2)<br>MW9B-GW-90-100-03-083021 (L35586-3 (A)) | PFDoS | ng/L | UJ | ms |
| MW9B-GW-90-100-01-083021 (L35586-1)<br>MW9B-GW-90-100-02-083021 (L35586-2)<br>MW9B-GW-90-100-03-083021 (L35586-3 (A)) | PFBA | ng/L | J | fd,ms |
| MW9B-GW-90-100-01-083021 (L35586-1)<br>MW9B-GW-90-100-02-083021 (L35586-2)<br>MW9B-GW-90-100-03-083021 (L35586-3 (A)) | PFPeA<br>PFHxA<br>PFBS<br>PFPeS | ng/L | J | fd |
| MW9B-GW-90-100-01-083021 (L35586-1) | PFOS | ng/L | J+ | q |
| MW14C-GW-16-36-01-090121 (L35586-4) | PFOS<br>PFOSA | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

c Calibration
fd Field duplicate
m Matrix spike
q Quantitation

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG78580 (WG78096)**

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 10/2/21 7:52 | NFDHA | 65.2% | 70-130% | NFDHA results were nondetect and qualified UJ. |
| CCV 10/6/21 18:57 | NFDHA | 66.3% | 70-130% | |
| CCV 10/6/21 22:00 | NFDHA | 65.5% | 70-130% | |

**MS/MSD Exceedances (Section 6Av):** The MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **OD5-WAT-BULK-03-083021:** | | | | | |
| PFBA | **156 / 155** | 70-130 | 0.16 | 30 | The detect for sample OD5-WAT-BULK-03-083021 was qualified as estimated biased high (J+). |
| PFDoS | 72.3 **/ 62.3** | 70-130 | 13.3 | 30 | The result for sample OD5-WAT-BULK-03-083021 was nondetect and qualified UJ. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

The parent and field duplicate (OD5-WAT-BULK-01-083021 and OD5-WAT-BULK-02-083021) were reported in DPWG78554 (WG78095). Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **OD5-WAT-BULK-01-083021 / OD5-WAT-BULK-02-083021 / OD5-WAT-BULK-02-083021** | | | | |
| PFHxA | 11 | 7.08 | 8.42 | 44.4 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP17C-WAT-BULK-01-083021 | L35587-20 i | N-EtFOSAA |
| MW14D-GW-6-21-01-083021 | L35587-32 | N-EtFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG78580 Report date (mm/dd/yyyy): 11/9/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/4/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at elevated temperatures of 6.2, 6.5, and 9.2 °C, and above the requirement of <6 °C. Results were not qualified, as the receipt temperatures were <10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | EQ-DIPPER-01-072821 (L35587-21 i)<br>EQ-AUGER-01-072821 (L35587-22 i)<br>EQ-PAIL-01-072821 (L35587-23 i)<br>EQ-TUBE-01-083021 (L35587-24 i)<br>EQ-DIPPER-01-083021 (L35587-33) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | No detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method MLA-110 limits used: 50% to 150% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OD5-WAT-BULK-03-083021 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - WAT-BULK-03-083021 (L35587-12)<br>FDs: OD5-WAT-BULK-01-083021 / OD5-WAT-BULK-02-083021 / OD5-WAT-BULK-03- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 083021 - Parent and FD reported in DPWG78554 (WG78095) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD for waters<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | The case narrative indicated that a number of samples were re-analyzed due to a mis-injection, and results were reported from the reanalysis. |

## 10. Additional comments about the lab report

**Reviewed using Method MLA-110MPCA limits and Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/4/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG78980 (WG78587)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 15.1 °C and did not meet the criteria of ≤6 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/8/21 16:02 | NFDHA | 50.8% | 70-130% | NFDHA results were not reported from this CCV. No qualifiers. |

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78587-101 | PFBS | 0.074 J | Associated detects within 10 times the method blank concentration were qualified B:<br><br>RC3C-SED-0-6-01-092121 (L35777-1)<br>RC3E-SED-0-6-01-092121 (L35777-3)<br>RC3F-SED-0-6-01-092121 (L35777-4)<br>RC3F-SED-0-6-02-092121 (L35777-5)<br>RC3F-SED-0-6-03-092121 (L35777-6 (A))<br>RC3G-SED-0-6-01-092121 (L35777-7)<br>RC3H-SED-0-6-01-092121 (L35777-8)<br>RC3I-SED-0-6-01-092121 (L35777-9)<br>RC3L-SED-0-6-01-092121 (L35777-12)<br>RC3L-SED-0-6-02-092121 (L35777-13)<br>RC3M-SED-0-6-01-092121 (L35777-14)<br>RC3N-SED-0-6-01-092121 (L35777-15)<br>RC3O-SED-0-6-01-092121 (L35777-16) |
| | PFOS | 0.069 J | The associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The result associated with the high recovery was qualified as estimated biased low (J-). Four results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3C-SED-0-6-01-092121 | L35777-1 | D3-N-MeFOSA | 35.4 | J+ |
| RC3C-SED-0-6-01-092121 | L35777-1 | D5-N-EtFOSA | 26.6 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3C-SED-0-6-01-092121 | L35777-1 | d9-NEt-FOSE | 39.8 | J+ |
| RC3D-SED-0-6-01-092121 | L35777-2 | D3-N-MeFOSA | 23.9 | J+ |
| RC3D-SED-0-6-01-092121 | L35777-2 | D5-N-EtFOSA | 18.7 | R |
| RC3D-SED-0-6-01-092121 | L35777-2 | D3-MeFOSAA | 36.7 | J+ |
| RC3D-SED-0-6-01-092121 | L35777-2 | d7-NMe-FOSE | 33.5 | UJ |
| RC3D-SED-0-6-01-092121 | L35777-2 | d9-NEt-FOSE | 24.2 | J+ |
| RC3E-SED-0-6-01-092121 | L35777-3 | D3-N-MeFOSA | 34.0 | J+ |
| RC3E-SED-0-6-01-092121 | L35777-3 | D5-N-EtFOSA | 24.3 | J+ |
| RC3E-SED-0-6-01-092121 | L35777-3 | d7-NMe-FOSE | 47.4 | UJ |
| RC3E-SED-0-6-01-092121 | L35777-3 | d9-NEt-FOSE | 32.0 | J+ |
| RC3F-SED-0-6-01-092121 | L35777-4 | 13C8-PFOSA | 203 | J- |
| RC3F-SED-0-6-01-092121 | L35777-4 | D5-N-EtFOSA | 41.1 | J+ |
| RC3F-SED-0-6-02-092121 | L35777-5 | D3-N-MeFOSA | 36.5 | J+ |
| RC3F-SED-0-6-02-092121 | L35777-5 | D5-N-EtFOSA | 25.8 | J+ |
| RC3F-SED-0-6-02-092121 | L35777-5 | D3-MeFOSAA | 31.9 | J+ |
| RC3F-SED-0-6-02-092121 | L35777-5 | d9-NEt-FOSE | 38.9 | J+ |
| RC3F-SED-0-6-03-092121 | L35777-6 (A) | D3-N-MeFOSA | 44.3 | J+ |
| RC3F-SED-0-6-03-092121 | L35777-6 (A) | D5-N-EtFOSA | 30.8 | J+ |
| RC3F-SED-0-6-03-092121 | L35777-6 (A) | D3-MeFOSAA | 35.2 | J+ |
| RC3F-SED-0-6-03-092121 | L35777-6 (A) | d9-NEt-FOSE | 48.0 | J+ |
| RC3G-SED-0-6-01-092121 | L35777-7 | D3-N-MeFOSA | 47.4 | J+ |
| RC3G-SED-0-6-01-092121 | L35777-7 | D5-N-EtFOSA | 39.7 | UJ |
| RC3H-SED-0-6-01-092121 | L35777-8 | 13C2-PFTeDA | 44.5 | J+<br>PFTrDA: J+ |
| RC3H-SED-0-6-01-092121 | L35777-8 | D3-N-MeFOSA | 37.2 | J+ |
| RC3H-SED-0-6-01-092121 | L35777-8 | D5-N-EtFOSA | 27.0 | UJ |
| RC3H-SED-0-6-01-092121 | L35777-8 | D3-MeFOSAA | 19.5 | R |
| RC3H-SED-0-6-01-092121 | L35777-8 | D5-EtFOSAA | 49.4 | J+ |
| RC3H-SED-0-6-01-092121 | L35777-8 | d9-NEt-FOSE | 45.3 | UJ |
| RC3I-SED-0-6-01-092121 | L35777-9 | D3-N-MeFOSA | 33.4 | J+ |
| RC3I-SED-0-6-01-092121 | L35777-9 | D5-N-EtFOSA | 24.4 | J+ |
| RC3I-SED-0-6-01-092121 | L35777-9 | D3-MeFOSAA | 44.1 | J+ |
| RC3I-SED-0-6-01-092121 | L35777-9 | d9-NEt-FOSE | 41.8 | J+ |
| RC3J-SED-0-6-01-092121 | L35777-10 | D3-N-MeFOSA | 38.1 | J+ |
| RC3J-SED-0-6-01-092121 | L35777-10 | D5-N-EtFOSA | 28.0 | J+ |
| RC3J-SED-0-6-01-092121 | L35777-10 | d9-NEt-FOSE | 46.5 | UJ |
| RC3K-SED-0-6-01-092121 | L35777-11 | D3-N-MeFOSA | 30.5 | J+ |
| RC3K-SED-0-6-01-092121 | L35777-11 | D5-N-EtFOSA | 20.2 | UJ |
| RC3K-SED-0-6-01-092121 | L35777-11 | d7-NMe-FOSE | 41.7 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3K-SED-0-6-01-092121 | L35777-11 | d9-NEt-FOSE | 32.3 | UJ |
| RC3L-SED-0-6-01-092121 | L35777-12 | D3-N-MeFOSA | 30.7 | J+ |
| RC3L-SED-0-6-01-092121 | L35777-12 | D5-N-EtFOSA | 21.9 | J+ |
| RC3L-SED-0-6-01-092121 | L35777-12 | D3-MeFOSAA | 42.2 | J+ |
| RC3L-SED-0-6-01-092121 | L35777-12 | d7-NMe-FOSE | 44.4 | UJ |
| RC3L-SED-0-6-01-092121 | L35777-12 | d9-NEt-FOSE | 33.4 | J+ |
| RC3L-SED-0-6-02-092121 | L35777-13 | 13C2-PFTeDA | 46.1 | J+<br>PFTrDA: J+ |
| RC3L-SED-0-6-02-092121 | L35777-13 | D3-N-MeFOSA | 26.1 | J+ |
| RC3L-SED-0-6-02-092121 | L35777-13 | D5-N-EtFOSA | 17.7 | R |
| RC3L-SED-0-6-02-092121 | L35777-13 | D3-MeFOSAA | 40.7 | J+ |
| RC3L-SED-0-6-02-092121 | L35777-13 | d7-NMe-FOSE | 32.8 | UJ |
| RC3L-SED-0-6-02-092121 | L35777-13 | d9-NEt-FOSE | 24.7 | J+ |
| RC3M-SED-0-6-01-092121 | L35777-14 | D3-N-MeFOSA | 31.7 | J+ |
| RC3M-SED-0-6-01-092121 | L35777-14 | D5-N-EtFOSA | 24.8 | J+ |
| RC3M-SED-0-6-01-092121 | L35777-14 | D3-MeFOSAA | 37.9 | J+ |
| RC3M-SED-0-6-01-092121 | L35777-14 | d9-NEt-FOSE | 41.2 | J+ |
| RC3N-SED-0-6-01-092121 | L35777-15 | D3-N-MeFOSA | 29.4 | J+ |
| RC3N-SED-0-6-01-092121 | L35777-15 | D5-N-EtFOSA | 21.7 | J+ |
| RC3N-SED-0-6-01-092121 | L35777-15 | d7-NMe-FOSE | 39.6 | UJ |
| RC3N-SED-0-6-01-092121 | L35777-15 | d9-NEt-FOSE | 33.4 | J+ |
| RC3O-SED-0-6-01-092121 | L35777-16 | D3-N-MeFOSA | 23.6 | J+ |
| RC3O-SED-0-6-01-092121 | L35777-16 | D5-N-EtFOSA | 17.0 | R |
| RC3O-SED-0-6-01-092121 | L35777-16 | D3-MeFOSAA | 30.3 | J+ |
| RC3O-SED-0-6-01-092121 | L35777-16 | d7-NMe-FOSE | 47.4 | UJ |
| RC3O-SED-0-6-01-092121 | L35777-16 | d9-NEt-FOSE | 34.8 | J+ |
| RC23A-SED-0-6-01-092221 | L35777-17 | 13C2-PFTeDA | 49.1 | J+<br>PFTrDA: J+ |
| RC23A-SED-0-6-01-092221 | L35777-17 | D3-N-MeFOSA | 43.6 | J+ |
| RC23A-SED-0-6-01-092221 | L35777-17 | D5-N-EtFOSA | 30.4 | UJ |
| RC23A-SED-0-6-01-092221 | L35777-17 | D3-MeFOSAA | 34.6 | J+ |
| RC23A-SED-0-6-01-092221 | L35777-17 | d7-NMe-FOSE | 47.3 | UJ |
| RC23A-SED-0-6-01-092221 | L35777-17 | d9-NEt-FOSE | 39.9 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC3F-SED-0-6-03-092121:** | | | | | |
| PFOA | **352/191** | 50-150 | 8.35 | 50 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **3060/6900** | 50-150 | 7.52 | 50 | |
| PFDS | 108/**164** | 50-150 | 8.37 | 50 | The detects for samples RC3F-SED-0-6-01-092121, RC3F-SED-0-6-02-092121, and RC3F-SED-0-6-03-092121 was qualified as estimated biased high (J+). |
| HFPO-DA | 130/83 | 50-150 | **52.2** | 50 | The result for sample RC3F-SED-0-6-03-092121 was nondetect, and was acceptable without qualification. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Field Triplicate Result (ng/g) | RPD (%) |
|---|---|---|---|---|
| **RC3F-SED-0-6-01-092121 / RC3F-SED-0-6-02-092121 / RC3F-SED-0-6-03-092121:** | | | | |
| PFBS | 0.621 B | 0.402 B | 0.758 | 60.0 |
| **RC3L-SED-0-6-01-092121 / RC3L-SED-0-6-02-092121:** | | | | |
| PFMPA | 0.516 | 1.17 | NA | 77.6 |
| PFBA | 6.94 | 12.1 | NA | 54.2 |
| PFBS | 0.30 B | 0.599 | NA | 66.5 |
| PFHpA | 0.683 | 1.31 | NA | 62.9 |
| PFOA | 7.15 | 13.5 | NA | 61.5 |
| PFPeS | 0.319 | 0.656 | NA | 69.1 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3C-SED-0-6-01-092121 | L35777-1 | N-MeFOSA |
| RC3G-SED-0-6-01-092121 | L35777-7 | PFDoS |
| RC3G-SED-0-6-01-092121 | L35777-7 | N-MeFOSA |
| RC3H-SED-0-6-01-092121 | L35777-8 | N-MeFOSA |
| RC3I-SED-0-6-01-092121 | L35777-9 | N-EtFOSA |
| RC3J-SED-0-6-01-092121 | L35777-10 | N-MeFOSA |
| RC3K-SED-0-6-01-092121 | L35777-11 | PFHpA |
| RC3K-SED-0-6-01-092121 | L35777-11 | N-MeFOSA |
| RC3L-SED-0-6-01-092121 | L35777-12 | N-EtFOSA |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3L-SED-0-6-01-092121 | L35777-12 | N-MeFOSA |
| RC3L-SED-0-6-02-092121 | L35777-13 | N-EtFOSA |
| RC3L-SED-0-6-02-092121 | L35777-13 | N-MeFOSA |
| RC3N-SED-0-6-01-092121 | L35777-15 | PFHxA |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG78980 Report date (mm/dd/yyyy): 11/19/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/10/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 15.1 °C and did not meet the criteria of ≤6 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFBS 0.074 J ng/g<br>PFOS 0.069 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC3F-SED-0-6-03-092121 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances and qualifications. |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs<br>FDs: RC3F-SED-0-6-01-092121 / RC3F-SED-0-6-02-092121 / RC3F-SED-0-6-03-092121<br>RC3L-SED-0-6-01-092121 / RC3L-SED-0-6-02-092121 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>MSD - RPDs: See data qualifications summary for exceedances.<br>FDs - See data qualifications summary for |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | Eight PFOS and two N-EtFOSAA results were reported from 5 times dilution analyses due to high sample concentrations. |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | The PFTeDA result for sample RC3H-SED-0-6-01-092121 (L35777-8) was reported from a reanalysis due to carry-over. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

**Four results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/10/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG78981 (WG78588)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 15.1 °C and did not meet the criteria of ≤6 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Seven results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC23B-SED-0-6-01-092221 | L35777-18 | 13C2-PFTeDA | 49.1 | J+<br>PFTrDA: J+ |
| RC23B-SED-0-6-01-092221 | L35777-18 | D3-N-MeFOSA | 37.8 | UJ |
| RC23B-SED-0-6-01-092221 | L35777-18 | D5-N-EtFOSA | 28.8 | UJ |
| RC23B-SED-0-6-01-092221 | L35777-18 | D3-MeFOSAA | 32.9 | J+ |
| RC23B-SED-0-6-01-092221 | L35777-18 | d7-NMe-FOSE | 42.6 | UJ |
| RC23B-SED-0-6-01-092221 | L35777-18 | d9-NEt-FOSE | 35.9 | UJ |
| RC23C-SED-0-6-01-092221 | L35777-19 | D3-N-MeFOSA | 49.3 | J+ |
| RC23C-SED-0-6-01-092221 | L35777-19 | D5-N-EtFOSA | 38.6 | J+ |
| RC23C-SED-0-6-01-092221 | L35777-19 | D3-MeFOSAA | 48.3 | J+ |
| RC22A-SED-0-6-01-092221 | L35777-20 | D3-N-MeFOSA | 33.7 | J+ |
| RC22A-SED-0-6-01-092221 | L35777-20 | D5-N-EtFOSA | 26.4 | UJ |
| RC22A-SED-0-6-01-092221 | L35777-20 | D3-MeFOSAA | 42.0 | J+ |
| RC22A-SED-0-6-01-092221 | L35777-20 | d9-NEt-FOSE | 41.4 | UJ |
| RC22B-SED-0-6-01-092221 | L35777-21 | D3-N-MeFOSA | 40.2 | UJ |
| RC22B-SED-0-6-01-092221 | L35777-21 | D5-N-EtFOSA | 28.1 | UJ |
| RC22B-SED-0-6-01-092221 | L35777-21 | D3-MeFOSAA | 40.0 | J+ |
| RC22B-SED-0-6-01-092221 | L35777-21 | d9-NEt-FOSE | 45.1 | UJ |
| RC23E-SED-0-6-01-092221 | L35777-22 | D5-N-EtFOSA | 39.1 | UJ |
| RC23F-SED-0-6-01-092221 | L35777-23 | 13C2-PFTeDA | 48.2 | J+<br>PFTrDA: J+ |
| RC23F-SED-0-6-01-092221 | L35777-23 | D3-N-MeFOSA | 31.6 | J+ |
| RC23F-SED-0-6-01-092221 | L35777-23 | D5-N-EtFOSA | 24.0 | J+ |
| RC23F-SED-0-6-01-092221 | L35777-23 | D3-MeFOSAA | 34.5 | J+ |
| RC23F-SED-0-6-01-092221 | L35777-23 | d7-NMe-FOSE | 48.0 | UJ |
| RC23F-SED-0-6-01-092221 | L35777-23 | d9-NEt-FOSE | 38.6 | UJ |
| RC4N-SED-0-6-01-092321 | L35777-25 | 13C2-PFDoA | 49.4 | J+ |
| RC4N-SED-0-6-01-092321 | L35777-25 | 13C2-PFTeDA | 32.2 | J+<br>PFTrDA: J+ |
| RC4N-SED-0-6-01-092321 | L35777-25 | D3-N-MeFOSA | 22.9 | J+ |
| RC4N-SED-0-6-01-092321 | L35777-25 | D5-N-EtFOSA | 18.0 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC4N-SED-0-6-01-092321 | L35777-25 | D3-MeFOSAA | 25.0 | J+ |
| RC4N-SED-0-6-01-092321 | L35777-25 | D5-EtFOSAA | 41.8 | J+ |
| RC4N-SED-0-6-01-092321 | L35777-25 | d7-NMe-FOSE | 34.3 | UJ |
| RC4N-SED-0-6-01-092321 | L35777-25 | d9-NEt-FOSE | 26.7 | UJ |
| RC4O-SED-0-6-01-092321 | L35777-26 | 13C2-PFDoA | 44.0 | J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | 13C2-PFTeDA | 25.5 | J+<br>PFTrDA: J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | 13C3-PFBS | 48.4 | J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | D3-N-MeFOSA | 20.4 | J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | D5-N-EtFOSA | 14.4 | R |
| RC4O-SED-0-6-01-092321 | L35777-26 | D3-MeFOSAA | 21.2 | J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | D5-EtFOSAA | 40.6 | J+ |
| RC4O-SED-0-6-01-092321 | L35777-26 | d7-NMe-FOSE | 35.1 | UJ |
| RC4O-SED-0-6-01-092321 | L35777-26 | d9-NEt-FOSE | 24.8 | UJ |
| RC4P-SED-0-6-01-092321 | L35777-27 | 13C2-PFTeDA | 44.6 | J+<br>PFTrDA: J+ |
| RC4P-SED-0-6-01-092321 | L35777-27 | D3-N-MeFOSA | 34.7 | J+ |
| RC4P-SED-0-6-01-092321 | L35777-27 | D5-N-EtFOSA | 26.8 | J+ |
| RC4P-SED-0-6-01-092321 | L35777-27 | D3-MeFOSAA | 29.5 | J+ |
| RC4P-SED-0-6-01-092321 | L35777-27 | d7-NMe-FOSE | 42.1 | UJ |
| RC4P-SED-0-6-01-092321 | L35777-27 | d9-NEt-FOSE | 34.8 | UJ |
| RC5B-SED-0-6-01-092321 | L35777-28 | D3-N-MeFOSA | 29.5 | J+ |
| RC5B-SED-0-6-01-092321 | L35777-28 | D5-N-EtFOSA | 23.1 | UJ |
| RC5B-SED-0-6-01-092321 | L35777-28 | D3-MeFOSAA | 29.4 | J+ |
| RC5B-SED-0-6-01-092321 | L35777-28 | d9-NEt-FOSE | 40.5 | UJ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | 13C4-PFHpA | 49.5 | J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | 13C8-PFOA | 49.1 | J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | 13C2-PFDoA | 47.8 | J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | 13C2-PFTeDA | 35.6 | J+<br>PFTrDA: J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | 13C3-PFHxS | 48.8 | J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | D3-N-MeFOSA | 18.9 | R |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | D5-N-EtFOSA | 14.9 | R |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | D3-MeFOSAA | 19.9 | R |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | D5-EtFOSAA | 37.5 | J+ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | d7-NMe-FOSE | 33.2 | UJ |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | d9-NEt-FOSE | 26.4 | UJ |
| RC5E-SED-0-6-01-092321 | L35777-30 | D3-N-MeFOSA | 43.6 | UJ |
| RC5E-SED-0-6-01-092321 | L35777-30 | D5-N-EtFOSA | 34.7 | UJ |
| RC5E-SED-0-6-01-092321 | L35777-30 | D3-MeFOSAA | 41.8 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC6C-SED-0-6-01-092321 | L35777-31 | 13C2-PFTeDA | 41.9 | J+<br>PFTrDA: J+ |
| RC6C-SED-0-6-01-092321 | L35777-31 | D3-N-MeFOSA | 24.5 | J+ |
| RC6C-SED-0-6-01-092321 | L35777-31 | D5-N-EtFOSA | 18.6 | R |
| RC6C-SED-0-6-01-092321 | L35777-31 | D3-MeFOSAA | 33.9 | J+ |
| RC6C-SED-0-6-01-092321 | L35777-31 | d7-NMe-FOSE | 39.5 | UJ |
| RC6C-SED-0-6-01-092321 | L35777-31 | d9-NEt-FOSE | 29.4 | UJ |
| RC6E-SED-0-6-01-092321 | L35777-32 | 13C2-PFTeDA | 34.3 | J+<br>PFTrDA: J+ |
| RC6E-SED-0-6-01-092321 | L35777-32 | D3-N-MeFOSA | 23.8 | UJ |
| RC6E-SED-0-6-01-092321 | L35777-32 | D5-N-EtFOSA | 18.3 | R |
| RC6E-SED-0-6-01-092321 | L35777-32 | D3-MeFOSAA | 47.5 | J+ |
| RC6E-SED-0-6-01-092321 | L35777-32 | d7-NMe-FOSE | 28.6 | UJ |
| RC6E-SED-0-6-01-092321 | L35777-32 | d9-NEt-FOSE | 22.4 | UJ |
| RC23G-SED-0-6-01-092421 | L35777-33 | 13C2-PFTeDA | 47.7 | UJ<br>PFTrDA: J+ |
| RC23G-SED-0-6-01-092421 | L35777-33 | D3-N-MeFOSA | 37.4 | J+ |
| RC23G-SED-0-6-01-092421 | L35777-33 | D5-N-EtFOSA | 29.3 | UJ |
| RC23G-SED-0-6-01-092421 | L35777-33 | D3-MeFOSAA | 29.0 | J+ |
| RC23G-SED-0-6-01-092421 | L35777-33 | d9-NEt-FOSE | 44.3 | UJ |
| RC23G-SED-0-6-02-092421 | L35777-34 | D3-N-MeFOSA | 43.9 | J+ |
| RC23G-SED-0-6-02-092421 | L35777-34 | D5-N-EtFOSA | 33.5 | UJ |
| RC23G-SED-0-6-02-092421 | L35777-34 | D3-MeFOSAA | 38.6 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Results did not require qualification due to MS/MSDs.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5B-SED-0-6-03-092321:** | | | | | |
| PFOS | 99.1/**227** | 50-150 | 1.86 | 50 | The sample concentration was greater than four times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **RC23G-SED-0-6-01-092421 / RC23G-SED-0-6-02-092421:** | | | |
| N-MeFOSAA | 0.173 | 0.086 J | 67.2 |
| PFDoA | 0.364 | 0.205 | 55.9 |
| PFDoS | 0.215 | 0.118 J | 58.3 |

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| PFDS | 1.73 | 0.903 | 62.8 |
| PFHpA | 0.346 | 0.191 | 57.7 |
| PFHpS | 0.462 | 0.242 | 62.5 |
| PFHxS | 0.29 | 0.166 | 54.4 |
| PFNS | 0.583 | 0.318 | 58.8 |
| PFOA | 5.96 | 3.35 | 56.1 |
| PFOS | 177 | 106 | 50.2 |
| PFOSA | 5.62 | 3.25 | 53.4 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC23B-SED-0-6-01-092221 | L35777-18 | N-MeFOSAA |
| RC23C-SED-0-6-01-092221 | L35777-19 | PFDoS |
| RC23E-SED-0-6-01-092221 | L35777-22 | N-MeFOSA |
| RC23E-SED-0-6-01-092221 | L35777-22 | PFDoS |
| RC23F-SED-0-6-01-092221 | L35777-23 | N-MeFOSA |
| RC4P-SED-0-6-01-092321 | L35777-27 | N-EtFOSA |
| RC5B-SED-0-6-03-092321 | L35777-29 (A) | N-MeFOSAA |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG78981 Report date (mm/dd/yyyy): 11/19/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/9/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 15.1 °C and did not meet the criteria of ≤6 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5B-SED-0-6-03-092321 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances. |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs<br>FDs: RC5B-SED-0-6-01-092321 / RC5B-SED-0-6-03-092321<br>RC23G-SED-0-6-01-092421 / RC23G-SED-0-6-02-092421 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☐ | ☒ | ☐ | MPCA limit: 50% RPD for solid (other)<br>MSD - RPDs ok<br>FDs: See data qualifications summary for exceedances. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Some of the samples were prepped using a lower sample weight. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | The PFOS results for samples RC4O-SED-0-6-01-092321 (L35777-26), RC4P-SED-0-6-01-092321 (L35777-27), and RC23G-SED-0-6-01-092421 (L35777-33) were reported from 3 times dilutions. |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

**Seven results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/10/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG78984 (WG78592)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 15.2 and15.9°C; and were above the 6 °C criteria. Detects for the samples were qualified as estimated biased low (J-) and nondetects were qualified as (UJ).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3C-WAT-Bulk-03-092121 | L35768-3 (A) | D3-MeFOSAA | 43.7 | UJ |
| RC3C-WAT-Bulk-01-092121 | L35768-5 | D3-MeFOSAA | 47.8 | UJ |
| RC3H-WAT-Bulk-01-092121 | L35768-6 | D3-MeFOSAA | 47.2 | UJ |
| RC23C-WAT-Bulk-01-092221 | L35768-7 | D3-MeFOSAA | 43.4 | UJ |
| RC22B-WAT-Bulk-01-092221 | L35768-8 | D3-MeFOSAA | 39.9 | UJ |
| RC20B-WAT-Bulk-01-092221 | L35768-9 | D3-MeFOSAA | 41.0 | UJ |
| RC4Q-WAT-Bulk-01-092321 | L35768-12 | D3-MeFOSAA | 44.3 | UJ |
| RC4K-WAT-Bulk-01-092321 | L35768-13 | D3-MeFOSAA | 38.7 | J+ |
| RC5A-WAT-Bulk-01-092321 | L35768-14 | D3-MeFOSAA | 42.3 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC3C-WAT-Bulk-03-092121:** | | | | | |
| PFHpA | **68.2**/108 | 70-130 | 17.6 | 30 | The detects for samples RC3C-WAT-Bulk-01-092121, RC3C-WAT-Bulk-02-092121, and RC3C-WAT-Bulk-03-092121 were qualified as estimated biased low (J-). |
| PFOA | **22.4/142** | 70-130 | 9.14 | 30 | The sample concentrations were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **423/197** | 70-130 | 4.16 | 30 | |
| NFDHA | **43.2**/89.7 | 70-130 | **69.4** | 30 | The results for samples RC3C-WAT-Bulk-01-092121, RC3C-WAT-Bulk-02-092121, and RC3C-WAT-Bulk-03-092121 were nondetect and qualified UJ. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **RC3C-WAT-Bulk-01-092121 / RC3C-WAT-Bulk-02-092121 / RC3C-WAT-Bulk-03-092121:** | | | | |
| PFBS | 6.53 | 8.71 | 6.41 | 31.9 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3H-WAT-Bulk-01-092121 | L35768-6 | PFDS |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG78984 Report date (mm/dd/yyyy): 11/22/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/11/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at elevated temperatures of 15.2 and 15.9 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | The trip blank field IDs and one sample ID were updated. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | EQ-Shovel-01-092221 (L35768-10) and EQ-Ziplock-01-092221 (L35768-11)<br>Trip blanks (L35768-1 and L35768-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC3C-WAT-Bulk-03-092121 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RC3C-WAT-Bulk-03-092121<br>FDs: RC3C-WAT-Bulk-01-092121 / RC3C-WAT-Bulk-02-092121 / RC3C-WAT-Bulk-03-092121 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | MPCA limit: 30% RPD for waters<br>See data qualifications summary for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/11/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79011 (WG78589)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 15.1 and 15.2 °C and did not meet the criteria of ≤6 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/6/21 14:47 | NFDHA | 60.1% | 70-130% | All NFDHA results were associated with this CCV and were qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Eight results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC23H-SED-0-6-01-092421 | L35777-35 | 13C2-PFTeDA | 37.5 | UJ<br>PFTrDA: J+ |
| RC23H-SED-0-6-01-092421 | L35777-35 | D3-N-MeFOSA | 33.7 | J+ |
| RC23H-SED-0-6-01-092421 | L35777-35 | D5-N-EtFOSA | 24.1 | J+ |
| RC23H-SED-0-6-01-092421 | L35777-35 | D3-MeFOSAA | 38.5 | J+ |
| RC23H-SED-0-6-01-092421 | L35777-35 | d7-NMe-FOSE | 46.4 | UJ |
| RC23H-SED-0-6-01-092421 | L35777-35 | d9-NEt-FOSE | 38.0 | UJ |
| RC26A-SED-0-6-01-092421 | L35777-36 | D3-N-MeFOSA | 41.8 | J+ |
| RC26A-SED-0-6-01-092421 | L35777-36 | D5-N-EtFOSA | 30.9 | UJ |
| RC26A-SED-0-6-01-092421 | L35777-36 | D3-MeFOSAA | 35.9 | J+ |
| RC26A-SED-0-6-01-092421 | L35777-36 | d9-NEt-FOSE | 47.4 | UJ |
| RC3I-SED-0-6-01-092421 | L35777-37 | 13C2-PFTeDA | 43.0 | J+<br>PFTrDA: J+ |
| RC3I-SED-0-6-01-092421 | L35777-37 | D3-N-MeFOSA | 19.5 | R |
| RC3I-SED-0-6-01-092421 | L35777-37 | D5-N-EtFOSA | 14.3 | R |
| RC3I-SED-0-6-01-092421 | L35777-37 | D3-MeFOSAA | 31.6 | J+ |
| RC3I-SED-0-6-01-092421 | L35777-37 | d7-NMe-FOSE | 31.6 | UJ |
| RC3I-SED-0-6-01-092421 | L35777-37 | d9-NEt-FOSE | 24.6 | UJ |
| RC3J-SED-0-6-01-092421 | L35777-38 | 13C2-PFTeDA | 46.1 | J+<br>PFTrDA: J+ |
| RC3J-SED-0-6-01-092421 | L35777-38 | D3-N-MeFOSA | 26.6 | J+ |
| RC3J-SED-0-6-01-092421 | L35777-38 | D5-N-EtFOSA | 16.4 | R |
| RC3J-SED-0-6-01-092421 | L35777-38 | D3-MeFOSAA | 33.4 | J+ |
| RC3J-SED-0-6-01-092421 | L35777-38 | d7-NMe-FOSE | 38.8 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3J-SED-0-6-01-092421 | L35777-38 | d9-NEt-FOSE | 28.9 | UJ |
| RC4Q-SED-0-6-01-092321 | L35777-39 | 13C2-PFTeDA | 46.3 | J+ PFTrDA: J+ |
| RC4Q-SED-0-6-01-092321 | L35777-39 | D3-N-MeFOSA | 15.1 | R |
| RC4Q-SED-0-6-01-092321 | L35777-39 | D5-N-EtFOSA | 10.1 | R |
| RC4Q-SED-0-6-01-092321 | L35777-39 | D3-MeFOSAA | 40.9 | J+ |
| RC4Q-SED-0-6-01-092321 | L35777-39 | d7-NMe-FOSE | 20.6 | UJ |
| RC4Q-SED-0-6-01-092321 | L35777-39 | d9-NEt-FOSE | 16.9 | R |
| RC4R-SED-0-6-01-092321 | L35777-40 | 13C2-PFTeDA | 49.8 | J+ PFTrDA: J+ |
| RC4R-SED-0-6-01-092321 | L35777-40 | D3-N-MeFOSA | 31.3 | J+ |
| RC4R-SED-0-6-01-092321 | L35777-40 | D5-N-EtFOSA | 21.7 | J+ |
| RC4R-SED-0-6-01-092321 | L35777-40 | D3-MeFOSAA | 41.0 | J+ |
| RC4R-SED-0-6-01-092321 | L35777-40 | d9-NEt-FOSE | 39.2 | J+ |
| RC4E-SED-0-6-01-092321 | L35777-41 | 13C2-PFTeDA | 49.6 | J+ PFTrDA: J+ |
| RC4E-SED-0-6-01-092321 | L35777-41 | D3-N-MeFOSA | 20.0 | J+ |
| RC4E-SED-0-6-01-092321 | L35777-41 | D5-N-EtFOSA | 13.6 | R |
| RC4E-SED-0-6-01-092321 | L35777-41 | D3-MeFOSAA | 33.8 | J+ |
| RC4E-SED-0-6-01-092321 | L35777-41 | d7-NMe-FOSE | 30.9 | UJ |
| RC4E-SED-0-6-01-092321 | L35777-41 | d9-NEt-FOSE | 23.8 | UJ |
| RC4F-SED-0-6-01-092321 | L35777-42 | 13C2-PFTeDA | 46.2 | J+ PFTrDA: J+ |
| RC4F-SED-0-6-01-092321 | L35777-42 | D3-N-MeFOSA | 26.7 | J+ |
| RC4F-SED-0-6-01-092321 | L35777-42 | D5-N-EtFOSA | 19.2 | R |
| RC4F-SED-0-6-01-092321 | L35777-42 | D3-MeFOSAA | 28.3 | J+ |
| RC4F-SED-0-6-01-092321 | L35777-42 | d7-NMe-FOSE | 37.1 | UJ |
| RC4F-SED-0-6-01-092321 | L35777-42 | d9-NEt-FOSE | 28.4 | UJ |
| RC4G-SED-0-6-01-092321 | L35777-43 | D3-N-MeFOSA | 37.3 | J+ |
| RC4G-SED-0-6-01-092321 | L35777-43 | D5-N-EtFOSA | 28.7 | J+ |
| RC4G-SED-0-6-01-092321 | L35777-43 | d9-NEt-FOSE | 48.0 | J+ |
| RC4H-SED-0-6-01-092321 | L35777-44 | D3-N-MeFOSA | 33.9 | J+ |
| RC4H-SED-0-6-01-092321 | L35777-44 | D5-N-EtFOSA | 24.9 | J+ |
| EP1C-SED-0-6-01-092721 | L35777-46 | D3-N-MeFOSA | 43.9 | UJ |
| EP1C-SED-0-6-01-092721 | L35777-46 | D5-N-EtFOSA | 34.2 | UJ |
| EP1E-SED-0-6-01-092721 | L35777-47 | D3-N-MeFOSA | 43.3 | UJ |
| EP1E-SED-0-6-01-092721 | L35777-47 | D5-N-EtFOSA | 33.6 | UJ |
| EP1D-SED-0-6-01-092721 | L35777-48 | D3-N-MeFOSA | 33.8 | UJ |
| EP1D-SED-0-6-01-092721 | L35777-48 | D5-N-EtFOSA | 25.9 | UJ |
| EP1D-SED-0-6-01-092721 | L35777-48 | D3-MeFOSAA | 46.9 | UJ |
| EP1D-SED-0-6-01-092721 | L35777-48 | d9-NEt-FOSE | 47.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17H-SED-0-6-01-092721 | L35777-49 | 13C2-PFTeDA | 45.6 | UJ<br>PFTrDA: UJ |
| EP17H-SED-0-6-01-092721 | L35777-49 | D3-N-MeFOSA | 36.1 | UJ |
| EP17H-SED-0-6-01-092721 | L35777-49 | D5-N-EtFOSA | 26.4 | UJ |
| EP17H-SED-0-6-01-092721 | L35777-49 | D3-MeFOSAA | 37.4 | J+ |
| EP17H-SED-0-6-01-092721 | L35777-49 | d9-NEt-FOSE | 44.4 | UJ |
| EP2D-SED-0-6-01-092821 | L35777-50 | D3-N-MeFOSA | 44.0 | UJ |
| EP2D-SED-0-6-01-092821 | L35777-50 | D5-N-EtFOSA | 34.2 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3I-SED-0-6-01-092421 | L35777-37 | PFDoS |
| RC3J-SED-0-6-01-092421 | L35777-38 | PFDoS |
| RC4R-SED-0-6-01-092321 | L35777-40 | PFBS |
| RC4G-SED-0-6-01-092321 | L35777-43 | PFBS |
| RC4G-SED-0-6-01-092321 | L35777-43 | PFHxA |
| EP1D-SED-0-6-01-092721 | L35777-48 | PFUnA |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79011 (WG78589) Report date (mm/dd/yyyy): 11/18/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/11/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 15.1 and 15.2°C and did not meet the criteria of ≤6 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Some of the samples IDs were updated. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances. |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD - RPDs ok<br>FDs: RC4H-SED-0-6-01-092321 / RC4H-SED-0-6-02-092321<br>EP2D-SED-0-6-01-092821 / EP2D-SED-0-6-02-092821 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other)<br>FDs:- RPDs ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Lower sample weights were used for samples RC4Q-SED-0-6-01-092321 (L35777-39) and RC4F-SED-0-6-01-092321 (L35777-42). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | The PFOS result for sample RC4F-SED-0-6-01-092321 (L35777-42) was reported from a 3 times dilutions due to a high sample concentration. |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

**Eight results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/12/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79012 (WG78590)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 15.3°C and did not meet the criteria of ≤6 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/6/21 14:47 Closing CCV | NFDHA | 60.1% | 70-130% | All NFDHA results were associated with this CCV and were qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP2D-SED-0-6-03-092821 | L35777-52 (A) | D5-N-EtFOSA | 45.9 | UJ |
| EP2D-SED-0-6-03-092821 | L35777-52 (A) | D3-MeFOSAA | 48.0 | UJ |
| EP2F-SED-0-6-01-092821 | L35777-53 | D3-N-MeFOSA | 41.4 | UJ |
| EP2F-SED-0-6-01-092821 | L35777-53 | D5-N-EtFOSA | 33.2 | UJ |
| EP2F-SED-0-6-01-092821 | L35777-53 | d9-NEt-FOSE | 44.6 | UJ |
| EP2G-SED-0-6-01-092821 | L35777-54 | D5-N-EtFOSA | 48.6 | UJ |
| EP17D-SED-0-6-01-092721 | L35777-55 | D5-N-EtFOSA | 38.9 | UJ |
| EP17F-SED-0-6-01-092721 | L35777-56 | 13C2-PFTeDA | 48.1 | UJ<br>PFTrDA: UJ |
| EP17F-SED-0-6-01-092721 | L35777-56 | D3-N-MeFOSA | 40.8 | J+ |
| EP17F-SED-0-6-01-092721 | L35777-56 | D5-N-EtFOSA | 33.3 | UJ |
| EP17F-SED-0-6-01-092721 | L35777-56 | d9-NEt-FOSE | 47.0 | UJ |
| EP17G-SED-0-6-01-092721 | L35777-57 | D3-N-MeFOSA | 45.6 | UJ |
| EP17G-SED-0-6-01-092721 | L35777-57 | D5-N-EtFOSA | 34.7 | UJ |
| EP17G-SED-0-6-01-092721 | L35777-57 | D3-MeFOSAA | 40.9 | J+ |
| EP17G-SED-0-6-01-092721 | L35777-57 | d9-NEt-FOSE | 47.6 | UJ |
| EP17M-WAT-Bulk-01-092721 | L35777-58 | 13C2-PFTeDA | 42.5 | UJ<br>PFTrDA: UJ |
| EP17M-WAT-Bulk-01-092721 | L35777-58 | D3-N-MeFOSA | 35.1 | J+ |
| EP17M-WAT-Bulk-01-092721 | L35777-58 | D5-N-EtFOSA | 26.4 | UJ |
| EP17M-WAT-Bulk-01-092721 | L35777-58 | d7-NMe-FOSE | 43.5 | UJ |
| EP17M-WAT-Bulk-01-092721 | L35777-58 | d9-NEt-FOSE | 34.1 | UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | 13C2-PFTeDA | 35.7 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | | | PFTrDA: UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | D3-N-MeFOSA | 32.5 | UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | D5-N-EtFOSA | 24.1 | UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | D3-MeFOSAA | 35.9 | UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | D5-EtFOSAA | 46.1 | J+ |
| EP18E-SED-0-6-01-092821 | L35777-59 | d7-NMe-FOSE | 35.4 | UJ |
| EP18E-SED-0-6-01-092821 | L35777-59 | d9-NEt-FOSE | 27.9 | UJ |
| EP18F-SED-0-6-01-092821 | L35777-60 | 13C2-PFTeDA | 41.2 | UJ<br>PFTrDA: J+ |
| EP18F-SED-0-6-01-092821 | L35777-60 | D3-N-MeFOSA | 33.6 | UJ |
| EP18F-SED-0-6-01-092821 | L35777-60 | D5-N-EtFOSA | 27.6 | UJ |
| EP18F-SED-0-6-01-092821 | L35777-60 | D3-MeFOSAA | 36.6 | UJ |
| EP18F-SED-0-6-01-092821 | L35777-60 | D5-EtFOSAA | 48.9 | J+ |
| EP18F-SED-0-6-01-092821 | L35777-60 | d7-NMe-FOSE | 34.4 | UJ |
| EP18F-SED-0-6-01-092821 | L35777-60 | d9-NEt-FOSE | 28.6 | UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | 13C2-PFTeDA | 38.5 | UJ<br>PFTrDA: UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | D3-N-MeFOSA | 33.8 | UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | D5-N-EtFOSA | 26.8 | UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | D3-MeFOSAA | 39.2 | UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | D5-EtFOSAA | 49.6 | J+ |
| EP26D-SED-0-6-01-092821 | L35777-61 | d7-NMe-FOSE | 45.8 | UJ |
| EP26D-SED-0-6-01-092821 | L35777-61 | d9-NEt-FOSE | 36.5 | UJ |
| EP26E-SED-0-6-01-092821 | L35777-62 | D5-N-EtFOSA | 46.3 | UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | 13C2-PFTeDA | 43.3 | UJ<br>PFTrDA: UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | D3-N-MeFOSA | 32.9 | UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | D5-N-EtFOSA | 26.3 | UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | D3-MeFOSAA | 47.2 | UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | d7-NMe-FOSE | 38.9 | UJ |
| EP26F-SED-0-6-01-092821 | L35777-63 | d9-NEt-FOSE | 34.4 | UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | 13C2-PFTeDA | 41.8 | UJ<br>PFTrDA: UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | D3-N-MeFOSA | 27.0 | UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | D5-N-EtFOSA | 23.0 | UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | D3-MeFOSAA | 36.2 | UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | d7-NMe-FOSE | 25.5 | UJ |
| EP26H-SED-0-6-01-092821 | L35777-64 | d9-NEt-FOSE | 23.2 | UJ |
| EP26K-SED-0-6-01-092821 | L35777-65 | 13C2-PFTeDA | 37.2 | UJ<br>PFTrDA: UJ |
| EP26K-SED-0-6-01-092821 | L35777-65 | D3-N-MeFOSA | 31.8 | UJ |
| EP26K-SED-0-6-01-092821 | L35777-65 | D5-N-EtFOSA | 22.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26K-SED-0-6-01-092821 | L35777-65 | D3-MeFOSAA | 46.1 | UJ |
| EP26K-SED-0-6-01-092821 | L35777-65 | d7-NMe-FOSE | 43.5 | UJ |
| EP26K-SED-0-6-01-092821 | L35777-65 | d9-NEt-FOSE | 32.6 | UJ |
| EP27F-SED-0-6-01-092821 | L35777-66 | 13C2-PFTeDA | 37.8 | UJ<br>PFTrDA: J+ |
| EP27F-SED-0-6-01-092821 | L35777-66 | D3-N-MeFOSA | 31.3 | UJ |
| EP27F-SED-0-6-01-092821 | L35777-66 | D5-N-EtFOSA | 23.7 | UJ |
| EP27F-SED-0-6-01-092821 | L35777-66 | D3-MeFOSAA | 38.1 | UJ |
| EP27F-SED-0-6-01-092821 | L35777-66 | d7-NMe-FOSE | 38.0 | UJ |
| EP27F-SED-0-6-01-092821 | L35777-66 | d9-NEt-FOSE | 31.6 | UJ |
| EP27J-SED-0-6-01-092821 | L35777-67 | D3-N-MeFOSA | 41.4 | UJ |
| EP27J-SED-0-6-01-092821 | L35777-67 | D5-N-EtFOSA | 30.2 | UJ |
| EP27J-SED-0-6-01-092821 | L35777-67 | d9-NEt-FOSE | 44.4 | UJ |
| EP27L-SED-0-6-01-092821 | L35777-69 | D3-N-MeFOSA | 40.8 | UJ |
| EP27L-SED-0-6-01-092821 | L35777-69 | D5-N-EtFOSA | 31.7 | UJ |
| EP27L-SED-0-6-01-092821 | L35777-69 | d9-NEt-FOSE | 40.7 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Results did not require qualification due to MS/MSDs.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP2D-SED-0-6-03-092821:** | | | | | |
| PFOS | **48.6/**54.0 | 50-150 | 0.736 | 50 | The sample concentration was greater than four times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP17D-SED-0-6-01-092721 | L35777-55 | PFBS |
| EP17F-SED-0-6-01-092721 | L35777-56 | PFBS |
| EP17F-SED-0-6-01-092721 | L35777-56 | N-MeFOSA |
| EP17G-SED-0-6-01-092721 | L35777-57 | PFBS |
| EP17I-SED-0-6-01-092721 | L35777-58 | N-MeFOSA |
| EP17I-SED-0-6-01-092721 | L35777-58 | N-MeFOSAA |
| EP18E-SED-0-6-01-092821 | L35777-59 | PFPeS |
| EP18F-SED-0-6-01-092821 | L35777-60 | N-EtFOSAA |
| EP26F-SED-0-6-01-092821 | L35777-63 | PFHxA |
| EP26H-SED-0-6-01-092821 | L35777-64 | PFDS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP26K-SED-0-6-01-092821 | L35777-65 | PFHxA |
| EP27F-SED-0-6-01-092821 | L35777-66 | PFHxA |
| EP27L-SED-0-6-01-092821 | L35777-69 | PFDS |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79012 (WG78590) Report date (mm/dd/yyyy): 11/22/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/12/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 15.3°C and did not meet the criteria of ≤6 °C. See data qualifications summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP2D-SED-0-6-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | MPCA PFAS other limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances. |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | FDs: EP27J-SED-0-6-01-092821 / EP27J-SED-0-6-02-092821 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | MPCA limit: 50% RPD for solid (other)<br>FDs:- RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Lower sample weights were used for samples EP18E-SED-0-6-01-092821 (L35777-59), EP18F-SED-0-6-01-092821 (L35777-60), and EP26H-SED-0-6-01-092821 (L35777-64). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | The PFOS result for sample RC4F-SED-0-6-01-092321 (L35777-42) was reported from a 3 times dilutions due to a high sample concentration. |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/12/2021

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79163 (WG78602)**

**Chain of custody preservation, and holding time (Section 1C):** 17 of the samples (all samples except sample EP18F-CTS-03-092821 [L35803-1]) were received at a temperature of 14.5 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ as they were received at a temperature ≥ 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Ten results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3C-GBR-01-092121 | L35758-1 | D3-MeFOSAA | 46.6 | UJ |
| RC3C-OVG-01-092121 | L35758-2 | d9-NEt-FOSE | 7.41 | R |
| RC3E-OVG-01-092121 | L35758-3 | d9-NEt-FOSE | 35.6 | UJ |
| RC3F-CTS-01-092121 | L35758-4 | d9-NEt-FOSE | 34.4 | UJ |
| RC3F-CTR-01-092121 | L35758-5 | D5-EtFOSAA | 47.0 | J+ |
| RC3F-CTR-01-092121 | L35758-5 | d9-NEt-FOSE | 9.24 | R |
| RC3G-CTR-01-092121 | L35758-6 | D5-EtFOSAA | 48.5 | UJ |
| RC3G-CTR-01-092121 | L35758-6 | d9-NEt-FOSE | 13.5 | R |
| RC3G-CTS-01-092121 | L35758-7 | D3-MeFOSAA | 45.9 | UJ |
| RC3G-CTS-01-092121 | L35758-7 | D5-EtFOSAA | 45.6 | UJ |
| RC3G-CTS-01-092121 | L35758-7 | d9-NEt-FOSE | 8.51 | R |
| RC3H-CTR-01-092121 | L35758-8 | d9-NEt-FOSE | 13.1 | R |
| RC3H-CTS-01-092121 | L35758-9 | d9-NEt-FOSE | 12.5 | R |
| RC3I-OVG-01-092121 | L35758-10 | 13C2-8:2 FTS | 47.3 | UJ |
| RC3I-OVG-01-092121 | L35758-10 | D3-MeFOSAA | 39.8 | UJ |
| RC3I-OVG-01-092121 | L35758-10 | D5-EtFOSAA | 46.2 | UJ |
| RC3K-CTS-01-092121 | L35758-11 | d9-NEt-FOSE | 9.8 | R |
| RC3K-CTR-01-092121 | L35758-12 | d9-NEt-FOSE | 14.0 | R |
| RC3J-GBR-01-092121 | L35758-13 | D3-MeFOSAA | 45.8 | UJ |
| RC3J-GBR-01-092121 | L35758-13 | D5-EtFOSAA | 49.4 | J+ |
| RC3M-GBR-01-092121 | L35758-15 | d9-NEt-FOSE | 16.7 | R |
| RC3N-GBR-01-092121 | L35758-16 | d9-NEt-FOSE | 28.6 | J+ |
| EP18F-CTS-03-092821 | L35803-1 (A) | D3-MeFOSAA | 47.1 | UJ |
| EP18F-CTS-03-092821 | L35803-1 (A) | D5-EtFOSAA | 49.1 | UJ |
| EP18F-CTS-03-092821 | L35803-1 (A) | d9-NEt-FOSE | 15.0 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78602-102 | 5:3 FTCA | 165 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | N-MeFOSE | 250 | 70-130 | |
| | N-EtFOSE | 135 | 70-130 | Associated detects (listed below) were qualified as estimated (J), as both low and high bias are present.<br><br>RC3C-OVG-01-092121 (L35758-2)<br>RC3G-CTS-01-092121 (L35758-7)<br>RC3H-CTS-01-092121 (L35758-9)<br>RC3K-CTS-01-092121 (L35758-11)<br>RC3M-GBR-01-092121 (L35758-15)<br>RC3N-GBR-01-092121 (L35758-16) |
| | PFDoS | 48.9 | 70-130 | Associated detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP18F-CTS-03-092821:** | | | | | |
| 7:3 FTCA | **153**/143 | 50-150 | 9.47 | 50 | The results for sample EP18F-CTS-03-092821 were nondetect, and were acceptable without qualification. |
| N-EtFOSE | **232/234** | 50-150 | 1.84 | 50 | |
| NFDHA | **37.2/47.7** | 50-150 | 22.2 | 50 | The NFDHA result for sample EP18F-CTS-03-092821 was nondetect and qualified UJ. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3C-GBR-01-092121 | L35758-1 | PFHxS |
| RC3C-OVG-01-092121 | L35758-2 | PFHpS |
| RC3F-CTS-01-092121 | L35758-4 | PFHxS |
| RC3F-CTR-01-092121 | L35758-5 | N-EtFOSAA |
| RC3G-CTS-01-092121 | L35758-7 | PFHxS |
| RC3H-CTS-01-092121 | L35758-9 | PFHxS |
| RC3K-CTS-01-092121 | L35758-11 | PFHxS |
| RC3K-CTR-01-092121 | L35758-12 | N-EtFOSAA |
| RC3O-GBR-01-092121 | L35758-17 | PFBS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3O-GBR-01-092121 | L35758-17 | N-MeFOSAA |
| RC3O-GBR-01-092121 | L35758-17 | NFDHA |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79163 Report date (mm/dd/yyyy): 12/6/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/19/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 14.5 °C and 5.8 °C. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFUnA 0.178 J ng/g<br>PFTrDA 0.153 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | PFUnA and PFTrDA were not detected in the samples. |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | N-MeFOSE 250%<br>N-EtFOSE 135%<br>5:3 FTCA 165%<br>See data qualifications summary for exceedances and qualifications (attachment). |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | PFDoS 48.9%<br>See data qualifications summary for exceedances and qualifications (attachment). |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP18F-CTS-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances (attachment). |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>No field duplicates |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☒ | ☐ | ☐ | 50% RPD limit used for tissue<br>MSD - RPDs ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Sample RC3F-CTR-01-092121 (L35758-5) was re-analyzed for PFBA due to possible sample carry-over. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Ten results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/20/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79164 (WG78603)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at temperatures of 14.1 and 14.5 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ as they were received at a temperature ≥ 10 °C.

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78603-101 | PFOS | 0.121 J | Associated detects that were within 10 times the method blank concentration, taking into consideration sample size, were qualified B:<br><br>RC3D-CTS-01-092121 (L35758-30) |
| | PFTeDA | 0.142 J | The associated results were nondetect. |
| | PFUnA | 0.137 J | |
| | PFTrDA | 0.149 J | Associated detects within 10 times the method blank concentration were qualified B:<br><br>RC22A-GBR-01-092221 (L35758-22) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Five results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3D-CTR-01-092121 | L35758-18 | d9-NEt-FOSE | 46.4 | UJ |
| RC3D-CTR-02-092121 | L35758-19 | d9-NEt-FOSE | 18.1 | R |
| RC20B-OVG-01-092221 | L35758-20 | D3-MeFOSAA | 49.9 | UJ |
| RC20B-OVG-01-092221 | L35758-20 | d9-NEt-FOSE | 8.86 | R |
| RC22B-OVG-01-092221 | L35758-21 | D3-MeFOSAA | 47.0 | UJ |
| RC22B-OVG-01-092221 | L35758-21 | d9-NEt-FOSE | 25.9 | UJ |
| RC22A-GBR-01-092221 | L35758-22 | D3-MeFOSAA | 46.2 | UJ |
| RC22A-GBR-01-092221 | L35758-22 | D5-EtFOSAA | 49.9 | J+ |
| RC22A-GBR-01-092221 | L35758-22 | d9-NEt-FOSE | 48.7 | UJ |
| RC23B-OVG-01-092221 | L35758-24 | d9-NEt-FOSE | 29.2 | UJ |
| RC23C-FAV-01-092221 | L35758-25 | D3-MeFOSAA | 48.6 | UJ |
| RC23E-FAV-01-092221 | L35758-27 | D3-MeFOSAA | 49.4 | UJ |
| RC23E-FAV-03-092221 | L35758-28 (A) | D3-MeFOSAA | 46.0 | UJ |
| RC3D-CTS-01-092121 | L35758-30 | d9-NEt-FOSE | 16.7 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC4P-CTS-01-092321 | L35758-31 | d9-NEt-FOSE | 38.7 | UJ |
| RC4P-CTR-01-092321 | L35758-32 | D5-EtFOSAA | 49.9 | J+ |
| RC4P-CTR-01-092321 | L35758-32 | d9-NEt-FOSE | 11.9 | R |
| RC5B-CTS-01-092321 | L35758-33 | D3-MeFOSAA | 45.8 | UJ |
| RC5B-CTS-01-092321 | L35758-33 | d9-NEt-FOSE | 43.3 | UJ |
| RC5B-CTR-01-092321 | L35758-34 | d9-NEt-FOSE | 13.1 | R |
| RC5B-CTS-02-092321 | L35758-35 | D3-MeFOSAA | 47.3 | UJ |
| RC5B-CTS-02-092321 | L35758-35 | d9-NEt-FOSE | 20.9 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78603-102 | N-MeFOSE | 209 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | NFDHA | 54.0 | 70-130 | The associated results were nondetect and qualified UJ. |
| | PFDoS | 56.1 | 70-130 | Associated detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |
| | PFTrDA | 133 | 70-130 | Associated detect (listed below) were qualified as estimated (J), as both low and high bias are present.<br><br>RC23C-FAV-01-092221 (L35758-25) |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC23E-FAV-03-092221:** | | | | | |
| 5:3 FTCA | 129/**189** | 50-150 | 38.9 | 50 | The results for sample RC23E-FAV-03-092221 were nondetect, and were acceptable without qualification. |
| 7:3 FTCA | 125/**165** | 50-150 | 28.7 | 50 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **RC3D-CTR-01-092121 / RC3D-CTR-02-092121:** | | | |
| PFOS | 9.59 | 22.1 | 79 |
| PFOSA | 0.60 | 2.38 | >2xRL |
| EtFOSAA | 0.39 J | 2.07 | >2xRL |
| **RC23E-FAV-01-092221 / RC23E-FAV-03-092221:** | | | |
| PFBA | 5.02 | 2.78 | 57.4 |
| PFOA | 0.718 | 0.395 | 58.0 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3D-CTR-01-092121 | L35758-18 | PFHxA |
| RC3D-CTR-01-092121 | L35758-18 | PFNS |
| RC3D-CTR-02-092121 | L35758-19 | N-MeFOSAA |
| RC20B-OVG-01-092221 | L35758-20 | PFBS |
| RC20B-OVG-01-092221 | L35758-20 | PFHxA |
| RC20B-OVG-01-092221 | L35758-20 | PFHxS |
| RC22A-GBR-01-092221 | L35758-22 | PFDoA |
| RC23B-OVG-01-092221 | L35758-24 | PFHxS |
| RC23C-FAV-01-092221 | L35758-25 | PFDA |
| RC23C-FAV-01-092221 | L35758-25 | PFTrDA |
| RC23D-FAV-01-092221 | L35758-26 | PFHxA |
| RC23D-FAV-01-092221 | L35758-26 | PFHxS |
| RC23E-FAV-01-092221 | L35758-27 | PFDA |
| RC23E-FAV-01-092221 | L35758-27 | PFHxS |
| RC23E-FAV-03-092221 | L35758-28 (A) | PFDA |
| RC23E-FAV-03-092221 | L35758-28 (A) | PFHxS |
| RC23F-FAV-01-092221 | L35758-29 | PFHxS |
| RC3D-CTS-01-092121 | L35758-30 | PFHxS |
| RC4P-CTS-01-092321 | L35758-31 | PFHxS |
| RC4P-CTR-01-092321 | L35758-32 | PFHxS |
| RC5B-CTS-01-092321 | L35758-33 | PFHxS |
| RC5B-CTS-02-092321 | L35758-35 | PFHxS |

Results with both high bias (J+) and low bias (J-) were qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79164 Report date (mm/dd/yyyy): 12/2/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/20/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 14.1 °C and 14.5 °C. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOS 0.121 J ng/g<br>PFTeDA 0.142 J ng/g<br>PFTrDA 0.149 J ng/g<br>PFUnA 0.137 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications (attachment). |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC23E-FAV-03-092221 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances (attachment). |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FDs: RC3D-CTR-01-092121 / RC3D-CTR-02-092121<br>RC23E-FAV-01-092221 / RC23E-FAV-03-092221<br>RC5B-CTS-01-092321 / RC5B-CTS-02- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 092321 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPDs ok<br>FDs - See data qualifications summary for exceedances and qualifications (attachment). |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Five results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/20/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79165 (WG78604)**

**Chain of custody preservation, and holding time (Section 1C):** All samples except EP18F-CTR-03-092821 (L35803-2) were received at a temperature of 14.1 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ as they were received at a temperature ≥ 10 °C.

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78604-101 | PFOSA | 0.144 J | Associated detects that were within 10 times the method blank concentration, taking into consideration sample size, were qualified B:<br><br>RC5C-GBR-01-092321 (L35758-36)<br>RC5D-OVG-01-092321 (L35758-37)<br>RC5E-CTR-01-092321 (L35758-39)<br>RC6E-CTR-01-092321 (L35758-47)<br>RC23H-FAV-01-092421 (L35758-50)<br>RC26A-GBR-01-092421 (L35758-52) |
| | PFUnA | 0.188 J | Associated detects were within 10 times the method blank concentration and were qualified B:<br><br>RC23I-FAV-01-092421 (L35758-51) |
| | PFMBA | 0.158 J | The associated results were nondetect. |
| | PFTrDA | 0.172 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Three results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC5C-GBR-01-092321 | L35758-36 | d9-NEt-FOSE | 39.6 | UJ |
| RC5D-OVG-01-092321 | L35758-37 | d9-NEt-FOSE | 7.42 | R |
| RC5E-CTS-01-092321 | L35758-38 | D3-MeFOSAA | 48.1 | UJ |
| RC5E-CTR-01-092321 | L35758-39 | d9-NEt-FOSE | 10.8 | R |
| RC5F-GBR-01-092321 | L35758-40 | d9-NEt-FOSE | 21.8 | UJ |
| RC5G-OVG-02-092321 | L35758-42 | D5-N-EtFOSA | 42.7 | UJ |
| RC5G-OVG-02-092321 | L35758-42 | D3-MeFOSAA | 47.2 | UJ |
| RC5G-OVG-02-092321 | L35758-42 | D5-EtFOSAA | 45.3 | UJ |
| RC6C-CTS-01-092321 | L35758-43 | D3-MeFOSAA | 46.6 | UJ |
| RC6C-CTS-01-092321 | L35758-43 | D5-EtFOSAA | 48.9 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC6D-GBR-01-092321 | L35758-45 | D3-MeFOSAA | 48.2 | UJ |
| RC6D-GBR-01-092321 | L35758-45 | d9-NEt-FOSE | 13.3 | R |
| RC6E-CTS-01-092321 | L35758-46 | D3-MeFOSAA | 48.7 | UJ |
| RC6E-CTS-01-092321 | L35758-46 | d9-NEt-FOSE | 27.8 | UJ |
| RC23G-GBR-02-092421 | L35758-49 | D5-EtFOSAA | 49.9 | UJ |
| RC23G-GBR-02-092421 | L35758-49 | d9-NEt-FOSE | 44.1 | UJ |
| RC23H-FAV-01-092421 | L35758-50 | 13C2-PFTeDA | 48.0 | UJ |
| RC23I-FAV-01-092421 | L35758-51 | d9-NEt-FOSE | 47.8 | UJ |
| EP18F-CTR-03-092821 | L35803-2 (A) | d9-NEt-FOSE | 26.0 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78604-102 | 5:3 FTCA | 241 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | 7:3 FTCA | 161 | 70-130 | |
| | N-MeFOSE | 219 | 70-130 | |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP18F-CTR-03-092821:** | | | | | |
| 5:3 FTCA | **157**/134 | 50-150 | 15.3 | 50 | The results for sample EP18F-CTR-03-092821 were nondetect, and were acceptable without qualification. |
| HFPO-DA | 146/85.9 | 50-150 | **51.1** | 50 | |
| N-EtFOSE | **257/229** | 50-150 | 11.1 | 50 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **RC5G-OVG-01-092321 / RC5G-OVG-02-092321:** | | | |
| 6:2 FTS | 9.38 | 0.707 U | >2xRL |
| **RC23G-GBR-01-092421 / RC23G-GBR-02-092421:** | | | |
| PFBA | 3.13 | 6.04 | 63.5 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC5D-OVG-01-092321 | L35758-37 | PFHxS |
| RC5D-OVG-01-092321 | L35758-37 | PFBS |
| RC6C-CTS-01-092321 | L35758-43 | PFHxS |
| RC6D-GBR-01-092321 | L35758-45 | PFHxS |
| RC6E-CTS-01-092321 | L35758-46 | PFHxS |
| RC5D-OVG-01-092321 | L35758-37 | PFHxS |
| RC5D-OVG-01-092321 | L35758-37 | PFBS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79165 Report date (mm/dd/yyyy): 12/4/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/20/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 5.8 and 14.1 °C. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.144 J ng/g<br>PFMBA 0.158 J ng/g<br>PFTrDA 0.172 J ng/g<br>PFUnA 0.188 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP18F-CTR-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances (attachment). |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FDs: RC3D-CTR-01-092121 / RC3D-CTR-02-092121<br>RC23E-FAV-01-092221 / RC23E-FAV-03-092221<br>RC5B-CTS-01-092321 / RC5B-CTS-02- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 092321 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPD for HFPO-DA 51.1%<br>FDs - See data qualifications summary for exceedances and qualifications (attachment). |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Five results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/22/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79166 (WG78606)**

**Chain of custody preservation, and holding time (Section 1C):** Samples listed below were received at a temperature of 14.6 °C. Sample detects were qualified as estimated biased low (J-) and nondetects were qualified UJ as they were received at a temperature ≥ 10 °C.

| Sample ID | Lab ID |
|---|---|
| RC4J-FAV-01-092321 | L35758-71 |
| RC4M-FAV-01-092321 | L35758-72 |
| RC4N-CTS-01-092321 | L35758-73 |
| RC4N-CTR-01-092321 | L35758-74 |
| RC4O-GBR-01-092321 | L35758-75 |
| RC4L-FAV-01-092321 | L35758-76 |

**Method Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78606-101 | PFOSA | 0.102 J | Associated detects that were within 10 times the method blank concentration, taking into consideration sample size, were qualified B:<br><br>RC4J-FAV-01-092321 (L35758-71)<br>EP1B-FAV-01-092721 (L35758-77)<br>EP1D-CTR-01-092721 (L35758-81) |
| | PFUnA | 0.145 J | Associated detects were within 10 times the method blank concentration and were qualified B:<br><br>EP1B-FAV-01-092721 (L35758-77) |
| | PFTrDA | 0.107 J | The associated results were nondetect. |
| | PFTeDA | 0.106 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC4M-FAV-01-092321 | L35758-72 | d9-NEt-FOSE | 37.7 | UJ |
| RC4N-CTS-01-092321 | L35758-73 | D3-MeFOSAA | 40.2 | UJ |
| RC4N-CTS-01-092321 | L35758-73 | D5-EtFOSAA | 45.7 | UJ |
| RC4N-CTS-01-092321 | L35758-73 | d9-NEt-FOSE | 12.8 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC4O-GBR-01-092321 | L35758-75 | d9-NEt-FOSE | 32.3 | UJ |
| RC4L-FAV-01-092321 | L35758-76 | D3-MeFOSAA | 45.2 | UJ |
| RC4L-FAV-01-092321 | L35758-76 | d9-NEt-FOSE | 40.3 | UJ |
| EP1B-FAV-01-092721 | L35758-77 | D3-MeFOSAA | 46.2 | UJ |
| EP1C-CTR-01-092721 | L35758-79 | d9-NEt-FOSE | 15.6 | R |
| EP1D-CTS-01-092721 | L35758-80 | D3-MeFOSAA | 44.4 | UJ |
| EP1D-CTR-01-092721 | L35758-81 | D3-MeFOSAA | 49.8 | UJ |
| EP1D-CTR-01-092721 | L35758-81 | d9-NEt-FOSE | 19.1 | R |
| EP1E-OVG-01-092721 | L35758-82 | D3-MeFOSAA | 46.0 | UJ |
| EP1E-OVG-01-092721 | L35758-82 | D5-EtFOSAA | 49.4 | UJ |
| EP1E-OVG-01-092721 | L35758-82 | d9-NEt-FOSE | 9.17 | R |
| EP1F-OVG-01-092721 | L35758-83 | d9-NEt-FOSE | 38.5 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and the results qualified are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78606-102 | 5:3 FTCA | 150 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | N-EtFOSE | 148 | 70-130 | |
| | N-MeFOSE | 224 | 70-130 | |
| | PFDoS | 69.6 | 70-130 | Associated results were nondetect and qualified UJ. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP27H-OVG-03-092821:** | | | | | |
| 5:3 FTCA | **164/200** | 50-150 | 19.5 | 50 | The results for sample EP27H-OVG-03-092821 were nondetect, and were acceptable without qualification. |
| N-EtFOSE | 118/**229** | 50-150 | 27.6 | 50 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Parent samples for the field duplicates were reported in SDG DPWG79225 / WG78608. Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **EP2F-CTR-01-092821 / EP2F-CTR-02-092821:** | | | |
| PFOS | 6.48 | 13.8 | 72.2 |
| **EP2G-GBR-01-092821 / EP2G-GBR-02-092821:** | | | |
| PFOS | 1.25 | 0.645 | 63.8 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC4N-CTS-01-092321 | L35758-73 | PFHxS |
| RC4N-CTR-01-092321 | L35758-74 | PFHxS |
| RC4L-FAV-01-092321 | L35758-76 | N-EtFOSA |
| EP1D-CTS-01-092721 | L35758-80 | PFHxS |
| EP1E-OVG-01-092721 | L35758-82 | PFHxS |
| EP1G-GBR-01-092721 | L35758-84 | PFHxS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79166 Report date (mm/dd/yyyy): 12/3/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/22/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 5.8 and 14.1 °C. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.102 J ng/g<br>PFTeDA 0.107 J ng/g<br>PFTrDA 0.106 J ng/g<br>PFUnA 0.145 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP27H-OVG-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | See data qualifications summary for exceedances (attachment). |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FDs: EP2F-CTS-01-092821 / EP2F-CTS-02-092821<br>EP2F-CTR-01-092821 / EP2F-CTR-02-092821<br>EP2G-GBR-01-092821 / EP2G-GBR-02- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 092821<br>Parent samples reported in SDG DPWG79225_WG78608 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPDs ok<br>FDs - See data qualifications summary for exceedances and qualifications (attachment). |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Four results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/22/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79167 (WG78607)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/20/21 9:03 | NFDHA | 60.6% | 70-130% | NFDHA results were nondetect and qualified UJ. |

**Method Blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78607-101 | PFUnA | 0.128 J | The associated results were nondetect. |
| | N-EtFOSE | 0.826 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17E-GBR-01-092721 | L35758-89 | D5-N-EtFOSA | 42.5 | UJ |
| EP17E-GBR-01-092721 | L35758-89 | d7-NMe-FOSE | 43.6 | UJ |
| EP17E-GBR-01-092721 | L35758-89 | d9-NEt-FOSE | 37.0 | UJ |
| EP17F-OVG-01-092721 | L35758-90 | D5-N-EtFOSA | 48.1 | UJ |
| EP17F-OVG-01-092721 | L35758-90 | d7-NMe-FOSE | 42.9 | UJ |
| EP17F-OVG-01-092721 | L35758-90 | d9-NEt-FOSE | 40.3 | UJ |
| EP17D-OVG-01-092721 | L35758-91 | D3-N-MeFOSA | 42.3 | UJ |
| EP17D-OVG-01-092721 | L35758-91 | D5-N-EtFOSA | 32.1 | UJ |
| EP17D-OVG-01-092721 | L35758-91 | d7-NMe-FOSE | 39.9 | UJ |
| EP17D-OVG-01-092721 | L35758-91 | d9-NEt-FOSE | 38.3 | UJ |
| EP17G-GBR-01-092721 | L35758-92 | D3-N-MeFOSA | 35.3 | UJ |
| EP17G-GBR-01-092721 | L35758-92 | D5-N-EtFOSA | 26.5 | UJ |
| EP17G-GBR-01-092721 | L35758-92 | d7-NMe-FOSE | 29.8 | UJ |
| EP17G-GBR-01-092721 | L35758-92 | d9-NEt-FOSE | 19.8 | R |
| EP17H-CTS-01-092721 | L35758-93 | D3-N-MeFOSA | 35.8 | UJ |
| EP17H-CTS-01-092721 | L35758-93 | D5-N-EtFOSA | 30.9 | UJ |
| EP17H-CTS-01-092721 | L35758-93 | d7-NMe-FOSE | 33.5 | UJ |
| EP17H-CTS-01-092721 | L35758-93 | d9-NEt-FOSE | 35.0 | UJ |
| EP17H-CTS-02-092721 | L35758-94 | D3-N-MeFOSA | 42.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17H-CTS-02-092721 | L35758-94 | D5-N-EtFOSA | 34.9 | UJ |
| EP17H-CTS-02-092721 | L35758-94 | d7-NMe-FOSE | 36.1 | UJ |
| EP17H-CTS-02-092721 | L35758-94 | d9-NEt-FOSE | 41.4 | UJ |
| EP17H-CTR-01-092721 | L35758-95 | D5-N-EtFOSA | 46.9 | UJ |
| EP17H-CTR-01-092721 | L35758-95 | d7-NMe-FOSE | 48.0 | UJ |
| EP17H-CTR-01-092721 | L35758-95 | d9-NEt-FOSE | 38.7 | UJ |
| EP17I-CTR-01-092721 | L35758-96 | D5-N-EtFOSA | 48.7 | UJ |
| EP17I-CTR-01-092721 | L35758-96 | d7-NMe-FOSE | 49.8 | UJ |
| EP17I-CTR-01-092721 | L35758-96 | d9-NEt-FOSE | 33.7 | UJ |
| EP17I-CTS-01-092721 | L35758-97 | 13C2-PFTeDA | 48.4 | UJ |
| EP17I-CTS-01-092721 | L35758-97 | D3-N-MeFOSA | 40.3 | UJ |
| EP17I-CTS-01-092721 | L35758-97 | D5-N-EtFOSA | 33.7 | UJ |
| EP17I-CTS-01-092721 | L35758-97 | d7-NMe-FOSE | 37.6 | UJ |
| EP17I-CTS-01-092721 | L35758-97 | d9-NEt-FOSE | 35.1 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | D3-N-MeFOSA | 48.5 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | D5-N-EtFOSA | 46.2 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | D3-MeFOSAA | 47.7 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | D5-EtFOSAA | 48.1 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | d7-NMe-FOSE | 47.7 | UJ |
| EP18E-CTR-02-092821 | L35758-99 | d9-NEt-FOSE | 47.8 | UJ |
| EP18E-GBR-03-092821 | L35758-100 (A) | D5-N-EtFOSA | 43.0 | UJ |
| EP18E-GBR-03-092821 | L35758-100 (A) | d7-NMe-FOSE | 42.7 | UJ |
| EP18E-GBR-03-092821 | L35758-100 (A) | d9-NEt-FOSE | 47.6 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | 13C2-8:2 FTS | 48.7 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | D3-N-MeFOSA | 48.6 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | D5-N-EtFOSA | 44.9 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | D3-MeFOSAA | 31.8 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | D5-EtFOSAA | 36.3 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | d7-NMe-FOSE | 41.6 | UJ |
| EP17K-FAV-01-092721 | L35803-5 | d9-NEt-FOSE | 36.9 | UJ |
| EP17L-FAV-01-092721 | L35803-6 | d7-NMe-FOSE | 49.0 | UJ |
| EP17L-FAV-01-092721 | L35803-6 | d9-NEt-FOSE | 42.2 | UJ |
| EP2D-GBR-01-092821 | L35803-7 | D3-N-MeFOSA | 45.7 | UJ |
| EP2D-GBR-01-092821 | L35803-7 | D5-N-EtFOSA | 41.3 | UJ |
| EP2D-GBR-01-092821 | L35803-7 | d7-NMe-FOSE | 40;0 | UJ |
| EP2D-GBR-01-092821 | L35803-7 | d9-NEt-FOSE | 43.9 | UJ |
| EP2D-GBR-02-092821 | L35803-8 | D3-N-MeFOSA | 44.6 | UJ |
| EP2D-GBR-02-092821 | L35803-8 | D5-N-EtFOSA | 40.4 | UJ |
| EP2D-GBR-02-092821 | L35803-8 | D3-MeFOSAA | 43.7 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP2D-GBR-02-092821 | L35803-8 | D5-EtFOSAA | 43.4 | UJ |
| EP2D-GBR-02-092821 | L35803-8 | d7-NMe-FOSE | 35.8 | UJ |
| EP2D-GBR-02-092821 | L35803-8 | d9-NEt-FOSE | 29.3 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and the results qualified are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78607-102 | 5:3 FTCA | 152 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | PFDoS | 44.6 | 70-130 | Associated results were nondetect and qualified UJ. |
| | N-MeFOSE | NQ | 70-130 | |

**Field Duplicates (Section 6Bi):**

Parent samples for field duplicates EP18E-CTR-02-092821 and EP18E-CTS-02-092821 were reported in SDG DPWG79225 / WG78608. Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **EP18E-CTR-01-092821 / EP18E-CTR-02-092821:** | | | |
| PFOS | 0.958 | 0.344 J | > 2xRL |
| **EP18E-CTS-01-092821 / EP18E-CTS-02-092821:** | | | |
| PFOS | 0.995 | 0.407 J | > 2xRL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP17F-OVG-01-092721 | L35758-90 | PFHxS |
| EP17H-CTS-02-092721 | L35758-94 | PFHxA |
| EP17H-CTR-01-092721 | L35758-95 | PFTrDA |
| EP17I-CTR-01-092721 | L35758-96 | PFTrDA |
| EP17I-CTS-01-092721 | L35758-97 | PFTrDA |
| EP17J-FAV-01-092721 | L35758-98 | PFTrDA |
| EP18E-GBR-03-092821 | L35758-100 (A) | PFHxS |
| EP18E-GBR-03-092821 | L35758-100 (A) | PFTrDA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79167 Report date (mm/dd/yyyy): 12/2/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/29/2022

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | The samples were received at temperatures of 5.8 and 8.2°C. Two of the samples included in SDG DPWG79167 were received at the laboratory at 8.2 °C. Results were not qualified as they were received at a temperature ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFUnA 0.128 J ng/g<br>N-EtFOSE 0.826 J ng/g<br>The laboratory indicated that the blank result for sample N-MeFOSE was not quantifiable. N-MeFOSE sample results were reported as nondetect. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP18E-GBR-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | PFAS other matrix limits used: 50% to 150%. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD<br>FDs: EP17H-CTS-01-092721 / EP17H-CTS-02-092721<br>EP18E-CTR-01-092821 / EP18E-CTR-02-092821<br>EP18E-CTS-01-092821 / EP18E-CTS-02-092821<br>EP2D-GBR-01-092821 / EP2D-GBR-02-092821<br>Some of the parent samples reported in SDG DPWG79225_WG78608 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPDs ok<br>FDs - See data qualifications summary for exceedances and qualifications (attachment). |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**One result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/30/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79168 (WG78609)**

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/20/21 19:34 | NFDHA | 68.2% | 70-130% | NFDHA results were nondetect and qualified UJ. |
| CCV 11/21/21 1:52 | ADONA | 134% | 70-130% | ADONA results were nondetect and were acceptable without qualification. |
| | NFDHA | 57.3% | 70-130% | NFDHA results were nondetect and qualified UJ. |

**Method Blank detections (Section 3Aii):** Method blank detections, and results qualified, are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78609-101 | PFOSA | 0.118 J | Sample detects were within 10 times the blank concentration and qualified BJ. |
| | PFUnA | 0.147 J | |
| | PFTrDA | 0.151 J | The associated results were nondetect. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Six results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26F-GBR-01-092821 | L35803-27 | d7-NMe-FOSE | 41.2 | UJ |
| EP26G-GBR-01-092821 | L35803-28 | D5-N-EtFOSA | 46.6 | UJ |
| EP26G-GBR-01-092821 | L35803-28 | d9-NEt-FOSE | 30.3 | UJ |
| EP26H-CTR-01-092821 | L35803-30 | d9-NEt-FOSE | 16.8 | R |
| EP26I-FAV-01-092821 | L35803-31 | d9-NEt-FOSE | 31.3 | UJ |
| EP26J-FAV-01-092821 | L35803-32 | D3-MeFOSAA | 42.8 | UJ |
| EP26J-FAV-01-092821 | L35803-32 | D5-EtFOSAA | 46.0 | J+ |
| EP26J-FAV-01-092821 | L35803-32 | d9-NEt-FOSE | 17.5 | R |
| EP26J-FAV-02-092821 | L35803-33 | D3-MeFOSAA | 48.5 | UJ |
| EP26J-FAV-02-092821 | L35803-33 | D5-EtFOSAA | 49.4 | J+ |
| EP26J-FAV-02-092821 | L35803-33 | d9-NEt-FOSE | 24.2 | UJ |
| EP26J-FAV-03-092821 | L35803-34 (A) | D3-MeFOSAA | 44.3 | UJ |
| EP26J-FAV-03-092821 | L35803-34 (A) | D5-EtFOSAA | 44.7 | J+ |
| EP26J-FAV-03-092821 | L35803-34 (A) | d9-NEt-FOSE | 22.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26K-CTS-01-092821 | L35803-35 | d9-NEt-FOSE | 44.8 | UJ |
| EP27D-OVG-01-092821 | L35803-37 | D5-N-EtFOSA | 34.4 | UJ |
| EP27D-OVG-01-092821 | L35803-37 | d9-NEt-FOSE | 5.58 | R |
| EP27E-OVG-01-092821 | L35803-38 | D3-MeFOSAA | 47.2 | UJ |
| EP27F-CTS-01-092821 | L35803-39 | D5-N-EtFOSA | 49.0 | UJ |
| EP27F-CTS-01-092821 | L35803-39 | d9-NEt-FOSE | 10.7 | R |
| EP27F-CTR-01-092821 | L35803-40 | d9-NEt-FOSE | 18.7 | R |
| EP27H-OVG-01-092821 | L35803-41 | 13C2-PFTeDA | 44.7 | UJ |
| EP27H-OVG-01-092821 | L35803-41 | D5-N-EtFOSA | 36.8 | UJ |
| EP27H-OVG-01-092821 | L35803-41 | D3-MeFOSAA | 46.8 | UJ |
| EP27J-GBR-01-092821 | L35803-43 | d9-NEt-FOSE | 42.9 | UJ |
| EP27K-OVG-01-092821 | L35803-44 | d9-NEt-FOSE | 42.9 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | 13C2-PFDoA | 44.2 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | 13C2-PFTeDA | 39.9 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | D3-N-MeFOSA | 46.7 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | D5-N-EtFOSA | 35.8 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | D3-MeFOSAA | 40.4 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | D5-EtFOSAA | 40.2 | UJ |
| EP27L-OVG-01-092821 | L35803-45 | d9-NEt-FOSE | 17.3 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78609-102 | 5:3 FTCA | 177 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | 7:3 FTCA | 146 | 70-130 | |
| | N-MeFOSE | 206 | 70-130 | |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP26J-FAV-03-092821:** | | | | | |
| 5:3 FTCA | **166/180** | 50-150 | 7.09 | 50 | The results for sample EP26J-FAV-03-092821 were nondetect, and were acceptable without qualification. |
| 9Cl-PF3ONS | **188/**138 | 50-150 | 30.9 | 50 | |
| 11Cl-PF3OUdS | **183/**135 | 50-150 | 30.4 | 50 | |
| ADONA | **200/**138 | 50-150 | 37.3 | 50 | |
| N-EtFOSE | **292/289** | 50-150 | 1.52 | 50 | |

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| | | | | | |
| PFOS | **245/**114 | 50-150 | 6.78 | 50 | The result for sample EP26J-FAV-03-092821 was greater than four times the spike concentration. No qualifiers. |
| NFDHA | **37.9/21.4** | 50-150 | **56.3** | 50 | The result for sample EP26J-FAV-03-092821 was nondetect and qualified UJ. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP26G-GBR-01-092821 | L35803-28 | PFHxS |
| EP26H-CTS-01-092821 | L35803-29 | PFHxS |
| EP26H-CTR-01-092821 | L35803-30 | 3:3 FTCA |
| EP26J-FAV-03-092821 | L35803-34 (A) | PFHxS |

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79168 Report date (mm/dd/yyyy): 12/6/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/30/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 8.2°C, and were above the ≤ 6 °C requirement. Results were not qualified as they were received at a temperature ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.118 J ng/g<br>PFUnA 0.147 J ng/g<br>PFTrDA 0.151 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP26J-FAV-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%<br>See data qualifications summary (attachment) for exceedances and qualifications.. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FDs: EP26J-FAV-01-092821 / EP26J-FAV-02-092821 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPD high for NFDHA (see attachment |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | for details)<br>FD - RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Six results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 1/30/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79225 (WG78608)**

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 11/20/21 9:03 | NFDHA | 60.7% | 70-130% | NFDHA results were nondetect and qualified UJ. |

**Method Blank detections (Section 3Aii):** Method blank detections, and results qualified, are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/g) | Qualifications |
|---|---|---|---|
| WG78608-101 | PFOSA | 0.104 J | The associated results were nondetect. |
| | PFTeDA | 0.14 J | |
| | PFUnA | 0.125 J | |
| | PFTrDA | 0.142 J | Sample detects were within 10 times the blank concentration and qualified BJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Five results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP2E-FAV-01-092821 | L35803-9 | D3-MeFOSAA | 38.4 | UJ |
| EP2E-FAV-01-092821 | L35803-9 | D5-EtFOSAA | 44.2 | UJ |
| EP2F-CTS-01-092821 | L35803-10 | d9-NEt-FOSE | 37.6 | UJ |
| EP2F-CTR-01-092821 | L35803-11 | d9-NEt-FOSE | 17.5 | R |
| EP2G-OVG-01-092821 | L35803-12 | D3-MeFOSAA | 49.4 | UJ |
| EP2G-OVG-01-092821 | L35803-12 | d9-NEt-FOSE | 6.98 | R |
| EP2G-GBR-01-092821 | L35803-13 | 13C5-PFHxA | 46.2 | J+ |
| EP2G-GBR-01-092821 | L35803-13 | 13C2-8:2 FTS | 40.6 | UJ |
| EP2G-GBR-01-092821 | L35803-13 | D3-MeFOSAA | 39.7 | UJ |
| EP2G-GBR-01-092821 | L35803-13 | D5-EtFOSAA | 40.4 | UJ |
| EP2G-GBR-01-092821 | L35803-13 | d7-NMe-FOSE | 48.8 | UJ |
| EP2G-GBR-01-092821 | L35803-13 | d9-NEt-FOSE | 24.0 | UJ |
| EP2H-GBR-01-092821 | L35803-14 | D3-MeFOSAA | 48.0 | UJ |
| EP2H-GBR-01-092821 | L35803-14 | D5-EtFOSAA | 47.7 | UJ |
| EP18E-CTS-01-092821 | L35803-15 | D3-MeFOSAA | 46.1 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18E-CTS-01-092821 | L35803-15 | D5-EtFOSAA | 49.7 | UJ |
| EP18E-CTR-01-092821 | L35803-16 | D3-MeFOSAA | 45.4 | UJ |
| EP18E-CTR-01-092821 | L35803-16 | D5-EtFOSAA | 46.6 | UJ |
| EP18E-GBR-01-092821 | L35803-17 | D3-MeFOSAA | 48.5 | UJ |
| EP18E-FAV-01-092821 | L35803-18 | D3-MeFOSAA | 47.0 | UJ |
| EP18E-FAV-01-092821 | L35803-18 | d9-NEt-FOSE | 27.3 | UJ |
| EP18E-FAV-02-092821 | L35803-19 | d9-NEt-FOSE | 49.0 | UJ |
| EP18G-OVG-01-092821 | L35803-20 | D3-MeFOSAA | 47.7 | UJ |
| EP18G-OVG-01-092821 | L35803-20 | d9-NEt-FOSE | 7.33 | R |
| EP18F-CTS-01-092821 | L35803-21 | D3-MeFOSAA | 49.2 | UJ |
| EP18F-CTS-01-092821 | L35803-21 | d9-NEt-FOSE | 12.4 | R |
| EP18F-CTR-01-092821 | L35803-22 | d9-NEt-FOSE | 29.9 | UJ |
| EP18F-GBR-01-092821 | L35803-23 | D5-EtFOSAA | 49.5 | UJ |
| EP18F-GBR-01-092821 | L35803-23 | d9-NEt-FOSE | 29.2 | UJ |
| EP26D-GBR-01-092821 | L35803-24 | d9-NEt-FOSE | 8.40 | R |
| EP26E-GBR-01-092821 | L35803-25 | d9-NEt-FOSE | 8.37 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances, and results qualified, are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG78608-102 | 5:3 FTCA | 133 | 70-130 | The associated results were nondetect, and were acceptable without qualification. |
| | N-MeFOSE | 249 | 70-130 | |
| | NFDHA | 65.9 | 70-130 | The associated results were nondetect and qualified UJ. |
| | PFDoS | 59.3 | 70-130 | |
| | N-EtFOSE | 137 | 70-130 | Associated detects were qualified as estimated biased high (J+). |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Results qualified are also discussed.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP26E-GBR-03-092821:** | | | | | |
| N-EtFOSE | **300/325** | 50-150 | 8.50 | 50 | The result for sample EP26E-GBR-03-092821 was nondetect, and was acceptable without qualification. |
| NFDHA | **57.5**/92.9 | 50-150 | 47.6 | 50 | The result for sample EP26E-GBR-03-092821 was nondetect and qualified UJ. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **EP26E-GBR-01-092821 / EP26E-GBR-03-092821:** | | | |
| PFOS | 9.34 | 5.59 | 50.2 |

Parent samples for the field duplicates listed below were reported in SDG DPWG79225 / WG78608, while field duplicates were reported in DPWG79166. Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **EP2F-CTR-01-092821 / EP2F-CTR-02-092821:** | | | |
| PFOS | 6.48 | 13.8 | 72.2 |
| **EP2G-GBR-01-092821 / EP2G-GBR-02-092821:** | | | |
| PFOS | 1.25 | 0.645 | 63.8 |

Parent samples for field duplicates EP18E-CTR-02-092821 and EP18E-CTS-02-092821 were reported in SDG DPWG79225 / WG78608, while field duplicates were reported in SDG DPWG79167. Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **EP18E-CTR-01-092821 / EP18E-CTR-02-092821:** | | | |
| PFOS | 0.958 | 0.344 J | > 2xRL |
| **EP18E-CTS-01-092821 / EP18E-CTS-02-092821:** | | | |
| PFOS | 0.995 | 0.407 J | > 2xRL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP2G-OVG-01-092821 | L35803-12 | PFHxS |
| EP2G-GBR-01-092821 | L35803-13 | PFHxA |
| EP2H-GBR-01-092821 | L35803-14 | PFHxS |
| EP18E-CTS-01-092821 | L35803-15 | PFHxS |
| EP18E-GBR-01-092821 | L35803-17 | PFHxS |
| EP18E-FAV-01-092821 | L35803-18 | PFHxS |
| EP18E-FAV-01-092821 | L35803-18 | PFUnA |
| EP18E-FAV-02-092821 | L35803-19 | PFUnA |
| EP18F-CTS-01-092821 | L35803-21 | PFHxS |
| EP18F-GBR-01-092821 | L35803-23 | PFTrDA |
| EP18F-GBR-01-092821 | L35803-23 | PFHxS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP26D-GBR-01-092821 | L35803-24 | PFHxS |
| EP26E-GBR-01-092821 | L35803-25 | PFHxS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79225 Report date (mm/dd/yyyy): 12/6/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/30/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 8.2°C, and were above the ≤ 6 °C requirement. Results were not qualified as they were received at a temperature ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.104 J ng/g<br>PFTeDA 0.14 J ng/g<br>PFUnA 0.125 J ng/g<br>PFTrDA 0.142 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Limits of 50% to 200% used for the tissue matrix. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP26E-GBR-03-092821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS other matrix limits used: 50% to 150%<br>See data qualifications summary (attachment) for exceedances and qualifications.. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FDs: EP18E-FAV-01-092821 / EP18E-FAV-02-092821<br>EP26E-GBR-01-092821 / EP26E-GBR-03-092821 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | Parent samples reported in this SDG, and field duplicate reported in:DPWG79166:<br>EP2F-CTS-01-092821 / EP2F-CTS-02-092821<br>EP2F-CTR-01-092821 / EP2F-CTR-02-092821<br>EP2G-GBR-01-092821 / EP2G-GBR-02-092821<br>Parent samples reported in this SDG, and field duplicate reported in:DPWG79167:<br>EP18E-CTR-01-092821 / EP18E-CTR-02-092821<br>EP18E-CTS-01-092821 / EP18E-CTS-02-092821 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit used for tissue<br>MSD - RPDs ok<br>FD - See data qualifications summary (attachment) for exceedances and qualifications.. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☒ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021), with the exception that acurracy and precision limits were adjusted for the tissue matrix.**

**See attachment for additional comments and qualifications.**

**Five results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/30/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79387 (WG78955)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ), as summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-110321 | L35965-1 | D5-N-EtFOSA | 49.2 | UJ |
| TRIP-COOLER3-110321 | L35965-3 | D3-N-MeFOSA | 48.1 | UJ |
| TRIP-COOLER3-110321 | L35965-3 | D5-N-EtFOSA | 49.6 | UJ |
| TRIP-COOLER4-110321 | L35965-4 | D5-N-EtFOSA | 48.4 | UJ |
| TRIP-COOLER5-110321 | L35965-5 | D5-N-EtFOSA | 49.4 | UJ |
| OD7-WAT-BULK-01-102921 | L35965-13 | d9-NEt-FOSE | 46.7 | UJ |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **OD4-WAT-BULK-01-102721 / OD4-WAT-BULK-02-102721 / OD4-WAT-BULK-03-102721:** | | | | |
| PFBA | 152 | 126 | 109 | 33.3 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79387 Report date (mm/dd/yyyy): 12/16/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 6.1, 7.2, 7.4, 7.9, 8.3 and 9.1°C, and were above the ≤ 6 °C criteria. Results were not qualified as they were received at a temperature ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Results for six trip blans reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TRIP-COOLER2-110321:<br>6:2 FTS 6.2 ng/L<br>TRIP-COOLER4-110321:<br>6:2 FTS 1.89 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | TRIP-COOLER1-110321 and TRIP-COOLER2-110321 were associated with the water samples reported in this SDG. 6:2 FTS results for the samples associated with TRIP-COOLER2-110321 were nondetect. No qualifiers. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OD4-WAT-BULK-03-102721 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | PFAS water limits used: 70% to 130% |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - ok<br>FD: OD4-WAT-BULK-01-102721 /<br>OD4-WAT-BULK-02-102721 / |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | OD4-WAT-BULK-03-102721 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>MSD - RPDs ok<br>FD - See data qualifications summary (attachment) for exceedances and qualifications.. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | RC3-WAT-BULK-01-102921 (SGS AXYS ID: L35965-17) was listed in this SDGs correlation table; however, the case narrative indicates it will be reported in a later SDG. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79388 (WG78956)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 12/1/21 3:43 Closing CCV | HFPO-DA | 63.8% | 70-130% | All HFPO-DA results were associated with this CCV and were qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ), as summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD7-WAT-BULK-03-102921 | L35965-14 (A) | D3-MeFOSAA | 47.8 | UJ |
| EP16-WAT-BULK-01-102921 | L35965-22 | d9-NEt-FOSE | 49.2 | UJ |
| EP17-WAT-BULK-01-102921 | L35965-23 | D3-MeFOSAA | 48.3 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **OD7-WAT-BULK-03-102921:** | | | |
| PFDoS | 63.5 | 70-130 | The PFDoS result for sample OD7-WAT-BULK-03-102921 was nondetect and qualified UJ. |
| PFHpA | 139 | 70-130 | The PFHpA detect for sample OD7-WAT-BULK-03-102921 was qualified as estimated biased high (J+). |
| PFOS | 296 | 70-130 | The sample concentration was greater than four times the spike concentration. No qualifiers. |
| 5:3 FTCA | 146 | 70-130 | The results for sample OD7-WAT-BULK-03-102921 were nondetect, and were acceptable without qualification. |
| ADONA | 131 | 70-130 | |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **RC5-WAT-BULK-01-102921 / RC5-WAT-BULK-02-102921:** | | | |
| PFDA | 8.84 | 5.11 | 53.48 |

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| PFOSA | 41.9 | 19.8 | 71.64 |
| EtFOSAA | 24.1 | 12.8 | 61.25 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79388 Report date (mm/dd/yyyy): 12/17/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/16/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 6.1, 7.2, and 8.3°C, and were above the ≤ 6 °C criteria. Results were not qualified as they were received at temperatures ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperatures are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were reported DPWG79387 (WG78955). TRIP-COOLER1-110321, TRIP-COOLER2-110321, and TRIP-COOLER3-110321 were associated with the water samples reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TRIP-COOLER2-110321:<br>6:2 FTS 6.2 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | 6:2 FTS results associated with TRIP-COOLER2-110321 were nondetect. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OD7-WAT-BULK-03-102921 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS water limits used: 70% to 130%<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | FD: RC5-WAT-BULK-01-102921 / RC5-WAT-BULK-02-102921 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data | | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>FD - See data qualifications summary (attachment) for exceedances and |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | The case narrative indicated that sample RC23-WAT-BULK-01-102921 and the MS was lost in the lab. PFAS results were not reported for sample 62664. The MSD was reported as the MS. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/16/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79508 (WG78957)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 12/6/21 22:28 Closing CCV | NFDHA | 54.5% | 70-130% | All NFDHA results were associated with this CCV and were qualified UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BL1-WAT-BULK-01-102821 | L35965-33 | 13C2-4:2 FTS | 171 | No Qualifier |
| BL1-WAT-BULK-01-102821 | L35965-33 | 13C2-PFTeDA | 30.9 | UJ PFTrDA: UJ |
| BL1-WAT-BULK-01-102821 | L35965-33 | d9-NEt-FOSE | 44.3 | UJ |
| CL1-WAT-BULK-02-102821 | L35965-36 | 13C2-4:2 FTS | 157 | No Qualifier |
| CL2-WAT-BULK-01-102821 | L35965-37 | d9-NEt-FOSE | 49.5 | UJ |
| TL1-WAT-BULK-01-102821 | L35965-39 | D5-N-EtFOSA | 49.9 | UJ |
| TL1-WAT-BULK-01-102821 | L35965-39 | d9-NEt-FOSE | 41.0 | UJ |
| TL2-WAT-BULK-01-102821 | L35965-40 | 13C4-PFBA | 36.3 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualifications are also listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **MW10C-GW-10-20-03-102821:** | | | |
| PFBA | **270/183** | 70-130 | The PFBA detects for samples MW10C-GW-10-20-01-102821, MW10C-GW-10-20-02-102821, and MW10C-GW-10-20-03-102821 were qualified as estimated biased high (J+). |
| NFDHA | **68.6**/86.4 | 70-130 | The NFDHA result for MW10C-GW-10-20-01-102821, MW10C-GW-10-20-02-102821, and MW10C-GW-10-20-03-102821 were nondetect and qualified UJ. |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **CL1-WAT-BULK-01-102821/CL1-WAT-BULK-02-102821:** | | | | |
| PFBA | 32.4 | 22.0 | -- | 38.2 |
| PFHxA | 3.46 | 2.28 | -- | 41.1 |
| PFOA | 2.9 | 1.76 | -- | 48.9 |
| PFTeDA | 4.86 | 3.34 | -- | 37.1 |
| **MW10C-GW-10-20-01-102821/MW10C-GW-10-20-02-102821/MW10C-GW-10-20-03-102821:** | | | | |
| PFBA | 1710 | 1680 | 960 | 51.7 |
| PFPeA | 35.2 | 32.1 | 18.3 | 59.2 |
| PFHxA | 29.6 | 24.1 | 17.4 | 51.5 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| CL2-WAT-BULK-01-102821 | L35965-37 | PFDA |
| PZAS-GW-6-16-01-102921 | L35965-44 | PFOA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79508 Report date (mm/dd/yyyy): 12/31/2021

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/22/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 6.1, 7.4, and 9.1°C, and were above the ≤ 6 °C criteria. Results were not qualified as they were received at temperatures ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were reported DPWG79387 (WG78955). TRIP-COOLER3-110321 (L35965-3), TRIP-COOLER4-110321 (L35965-4), and TRIP-COOLER5-110321 (L35965-5) were associated with the water samples reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TRIP-COOLER4-110321:<br>6:2 FTS 1.89 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | 6:2 FTS results associated with TRIP-COOLER4-110321 were nondetect. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW10C-GW-10-20-03-102821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS water limits used: 70% to 130%<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | FDs: CL1-WAT-BULK-01-102821/CL1-WAT-BULK-02-102821<br>MW10C-GW-10-20-01-102821/MW10C-GW-10-20-02-102821/MW10C-GW-10-20-03-102821 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Reduced sample volumes were used for 6 samples. The laboratory indicated that a reduced volumes were required due to high suspended solids content. The reduced sample volumes resulted in elevated reporting limits.<br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☐ | ☒ | ☐ | Reduced sample volumes for 6 samples were not discussed (see Section 7B). The laboratory was contacted, and they indicated that reduced sample volumes will be discussed in the case narratives going forward. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/22/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79511 (WG79105)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 12/6/21 22:28 Closing CCV | NFDHA | 54.3% | 70-130% | All NFDHA results were associated with this CCV and were qualified UJ. |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and the results qualified are listed below.

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG79105-102 | 11Cl-PF3OUdS | 74.8/129 | 70-130 | **53.5** | 30 | Associated results were nondetect and were acceptable without qualification. |
| | 4:2 FTS | 70.7/100 | 70-130 | **34.5** | 30 | |
| | 9Cl-PF3ONS | 84.2/**139** | 70-130 | **48.8** | 30 | |
| | ADONA | 86/**133** | 70-130 | **43.0** | 30 | |
| | HFPO-DA | 90/**141** | 70-130 | **43.9** | 30 | |
| | NFDHA | **47.1/39.9** | 70-130 | 16.5 | 30 | Associated results were nondetect and qualified UJ. |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC3-WAT-BULK-01-102921 | L35965-17 R | PFBS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79511 Report date (mm/dd/yyyy): 1/4/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/22/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperatures of 7.2°C, and above the ≤ 6 °C criteria. Results were not qualified as they were received at a temperature ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | The associated trip blank was reported in DPWG79387 (WG78955). TRIP-COOLER2-110321 (L35965-2) was associated with the water samples reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TRIP-COOLER2-110321:<br>6:2 FTS 6.62 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | 6:2 FTS results associated with TRIP-COOLER2-110321 were nondetect. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications.. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | MS/MSD results were not included in this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>See data qualifications summary (attachment) for exceedances and qualifications.. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See discussion in Section 9.<br><br>Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | The case narrative indicated that samples reported in DPWG79511/WG79105 were originally extracted in batches WG78955 and WG78956; however, extraction and analysis could not proceed as the samples could not pass through the SPE cartridges due to clogging. Samples were re-prepped with reduced volumes and reported in this SDG. The reduced volumes resulted in elevated reporting limits. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/22/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79512 (WG78958)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZCS-GW-6-11-01-102921 | L35965-49 | 13C2-PFTeDA | 48.0 | UJ<br>PFTrDA: UJ |
| PZCS-GW-6-11-01-102921 | L35965-49 | D5-N-EtFOSA | 46.0 | UJ |
| PZCS-GW-6-11-01-102921 | L35965-49 | d9-NEt-FOSE | 43.1 | UJ |
| PZED-GW-16-21-01-102621 | L35965-54 | D3-MeFOSAA | 49.3 | UJ |
| PZFD-GW-33-38-01-102821 | L35965-56 | d9-NEt-FOSE | 49.0 | UJ |
| PZGS-GW-20-30-01-102821 | L35965-57 | 13C2-PFTeDA | 44.4 | UJ<br>PFTrDA: UJ |
| PZGS-GW-20-30-01-102821 | L35965-57 | D5-N-EtFOSA | 44.1 | UJ |
| PZGS-GW-20-30-01-102821 | L35965-57 | d9-NEt-FOSE | 41.3 | UJ |
| PZGS-GW-20-30-02-102821 | L35965-58 | D3-N-MeFOSA | 45.5 | UJ |
| PZGS-GW-20-30-02-102821 | L35965-58 | D5-N-EtFOSA | 42.9 | UJ |
| PZGS-GW-20-30-02-102821 | L35965-58 | d9-NEt-FOSE | 43.1 | UJ |
| PZGS-GW-20-30-03-102821 | L35965-59 (A) | D5-N-EtFOSA | 47.6 | UJ |
| PZGS-GW-20-30-03-102821 | L35965-59 (A) | d9-NEt-FOSE | 45.6 | UJ |
| PZHD-GW-21-26-01-102921 | L35965-62 | D3-N-MeFOSA | 49.2 | UJ |
| PZHD-GW-21-26-01-102921 | L35965-62 | D5-N-EtFOSA | 45.7 | UJ |
| PZHD-GW-21-26-01-102921 | L35965-62 | d9-NEt-FOSE | 45.6 | UJ |
| TRIP-COOLER7-110321 | L35970-1 | D5-N-EtFOSA | 48.2 | UJ |
| TRIP-COOLER8-110321 | L35970-2 | D5-N-EtFOSA | 46.1 | UJ |
| TRIP-COOLER8-110321 | L35970-2 | d9-NEt-FOSE | 47,4 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualification was not required.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **PZGS-GW-20-30-03-102821:** | | | |
| PFBA | **57.5**/72.7 | 70-130 | The sample concentration was greater than four times the spike concentration. No qualifiers. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79512 Report date (mm/dd/yyyy): 1/10/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/28/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 6.1, 6.8, 7.4 and 7.9°C, and were above the ≤ 6 °C criteria. Results were not qualified as they were received at temperatures ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER7-110321 (L35970-1) and TRIP-COOLER8-110321 (L35970-2) reported in this SDG.<br>Associated trip blanks were reported DPWG79387 (WG78955). TRIP-COOLER5-110321 (L35965-5), and TRIP-COOLER6-110321 (L35965-6) were associated with the water samples reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZGS-GW-20-30-03-102821 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS water limits used: 70% to 130%<br>See data qualifications summary (attachment) for exceedances. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | FDs: PZGS-GW-20-30-01-102821/PZGS-GW-20-30-02-102821/PZGS-GW-20-30-03-102821 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/28/2022

**SGS AXYS DPWG79518** 4/30/22 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| WCLE-GW-0-93-01-110121 | PFOS | 10599 | 21177 | 1 | 62.56 | 62.6 | 0.0644 | 0.004 | L35974-4 |
| OP-Z-GW-145-185-01-110221 | PFOS | 5906 | 20166 | 1 | 37.07 | 37.1 | 0.064 | 0.004 | L35974-5 |
| LCS (WG79085-102) | PFOS | 15350 | 25527 | 1 | 4.84 | 4.84 | 1.00 | 0.004 | |
| LCS (WG79085-104) | PFOS | 13643 | 21524 | 1 | 5.10 | 5.1 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 12/3/21 21:02 | 22042 | 38574 | 4.600 | 4.6 | 5.00 | 92.0 | 92.0% |
| CCV 12/3/21 23:29 | 22530 | 38247 | 4.742 | 4.74 | 5.00 | 94.8 | 94.8% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG79085-101): | PFOS | 0.000 | <0.4 | verified <0.4 |

AECOM
1555 N. RiverCenter Drive, Suite 214
Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG79518 (Analysis WG79085) dated 1/5/2022 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 4/30/2022 |

The samples listed below were collected at the Project 1007 Site on October 4 and 5, and November 1 and 2, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| OW20J1-GW-86-90-01-100421 | | 10/4/21 | L35974-1 |
| OW20J1-GW-146-150-01-100521 | | 10/5/21 | L35974-2 |
| OW20J1-GW-146-150-02-100521 | Field Duplicate of OW20J1-GW-146-150-01-100521 | 10/5/21 | L35974-3 |
| WCLE-GW-0-93-01-110121 | | 11/1/21 | L35974-4 |
| OP-Z-GW-145-185-01-110221 | | 11/2/21 | L35974-5 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance document listed above, and the analytical method (MLA-110).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✗ Sample Receipt and Holding times
✗ Initial calibration/continuing calibration verification
✓ Laboratory and trip blanks
✗ Labeled Compounds
NA Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✗ Laboratory control sample (LCS)
✓ Field duplicates

✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to calibration verification, labeled compound recoveries, laboratory control samples, and quantitations as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact. The samples were received above 6 °C at a temperature of 13.7 °C. The reporting limits for nondetect results were qualified as estimated (UJ), and detects were qualified as estimated biased low (J-).

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for a number of analytes (and the two highest standards were excluded for NFDHA) from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification (SSV) recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 12/3/21 21:02 | NFDHA | 60.7% | 70-130% | NFDHA results were nondetect and qualified as estimated (UJ). |

Two CCVs were associated with this data set and results were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The chain of custody indicates the associated trip blank is TRIP-COOLER12-110321 (L365970-6). The database indicates the results for this trip blank were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, except as listed in that table below. Results associated with low recoveries were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW20J1-GW-86-90-01-100421 | L35974-1 | d7-NMe-FOSE | 49.8 | UJ |
| OW20J1-GW-86-90-01-100421 | L35974-1 | d9-NEt-FOSE | 43.8 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | 13C2-PFDoA | 40.7 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | 13C2-PFTeDA | 28.5 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | D3-N-MeFOSA | 34.7 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | D5-N-EtFOSA | 28.8 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | D3-MeFOSAA | 41.7 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | D5-EtFOSAA | 39.3 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | d7-NMe-FOSE | 27.5 | UJ |
| OW20J1-GW-146-150-01-100521 | L35974-2 | d9-NEt-FOSE | 24.2 | UJ |
| OW20J1-GW-146-150-02-100521 | L35974-3 | d9-NEt-FOSE | 44.0 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSDs were not associated with this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits except as listed in the table below.

| LCS IDs | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG79085-103 | N-MeFOSA | 62.8 | 70-130 | Associated results were nondetect and were qualified UJ. |
| | N-EtFOSA | 66.4 | 70-130 | |
| | NFDHA | 62.7 | 70-130 | |

**Field Duplicate Results**

Sample OW20J1-GW-146-150-02-100521 was collected as a field duplicate of OW20J1-GW-146-150-01-100521. RPDs were within the 30 percent limit for water sample, or results were within ± the limit of quantification.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for PFOS and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Reduced sample volume was used for WCLE-GW-0-93-01-110121 and OP-Z-GW-145-185-01-110221 due to high analyte concentrations. Reduced sample volumes were also used for sample OW20J1-GW-146-150-01-100521 and OW20J1-GW-146-150-02-100521; however, low concentrations were observed for these samples. The sample prep logs indicate that reduced sample volumes were used for samples WCLE-GW-0-93-01-110121 and OW20J1-GW-146-150-02-100521 due to high levels of particulates/solids in the samples.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| OW20J1-GW-86-90-01-100421 | L35974-1 | PFOSA | No | The results were qualified as estimated biased high (J+) as they included quantitation of an isomer that did not meet the signal to noise criteria. |
| OW20J1-GW-146-150-02-100521 | L35974-3 | PFOSA | No | |
| WCLE-GW-0-93-01-110121 | L35974-4 | PFOS | No | The results were qualified as estimated biased high (J+) as they included quantitation of a branched isomer that did not meet the ion ratio criteria. |
| OP-Z-GW-145-185-01-110221 | L35974-5 | PFOS | No | |

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high bias (J+) and low bias (J-) should be qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | All PFAS Analytes | ng/L | Detects: J- Nondetect: UJ | sr |
| All samples | NFDHA | ng/L | UJ | c,l |
| | N-MeFOSA<br>N-EtFOSA | ng/L | UJ | l |
| OW20J1-GW-86-90-01-100421 (L35974-1) | NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| OW20J1-GW-146-150-01-100521 (L35974-2) | PFDoA<br>PFTeDA<br>N-MeFOSA<br>N-EtFOSA<br>MeFOSAA<br>EtFOSAA<br>NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| OW20J1-GW-146-150-02-100521 (L35974-3) | NEt-FOSE | ng/L | UJ | s |
| OW20J1-GW-86-90-01-100421 (L35974-1)<br>OW20J1-GW-146-150-02-100521 (L35974-3) | PFOSA | ng/L | J | q,sr |
| WCLE-GW-0-93-01-110121 (L35974-4)<br>OP-Z-GW-145-185-01-110221 (L35974-5) | PFOS | ng/L | J | q,sr |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

l Laboratory control sample
q Quantitation
s Labeled compound
sr Sample receipt

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79725 (WG79331)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18E-FOAM-DRY-01-111821 | L36136-2 | D3-MeFOSAA | 46.2 | UJ |
| EP18E-FOAM-FROZEN-01-111821 | L36136-3 | D3-MeFOSAA | 46.7 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW10B-GW-116-120-03-112221:** | | | | | |
| PFDoS | 76.6/**68.0** | 70-130 | 14.0 | 30 | The PFDoS result for sample MW10B-GW-116-120-03-112221 was nondetect and qualified as estimated (UJ). |
| PFBA | **-63.9/29.7** | 70-130 | 18.6 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79725 Report date (mm/dd/yyyy): 1/20/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/23/2022

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-120121 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW10B-GW-116-120-03-112221 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | PFAS water limits used: 70% to 130%<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | The parent sample and field duplicate for MW10B-GW-116-120-03-112221 (L36136-4) are reported in SDG DPWG79727 (WG79331) and are evaluated in the data validation report for this SDG. |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix<br>RPD for MS/MSD -ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>A reduced sample volumes were used for samples EP18E-FOAM-DRY-01-111821 and EP18E-FOAM-FROZEN-01-111821 due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/24/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG79528 (WG78960)**

**Chain of custody preservation, and holding time (Section 1C):** Three samples (EQ-BLADDER-110121, EQ-TUBE-110121, and EQ-DIPPER-110121) were received at a temperature of 13.7 °C. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ, as they were received at a temperature >10 °C.

**Blank detections (Section 3Aii):** Equipment blank detections and the results qualified are listed in the table below.

| Equipment Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| EQ-DIPPER-110121 | 6:2 FTS | 6.31 J | Associated samples were not reported in this SDG. No qualifiers. |
| | PFBA | 2.51 J | |
| | PFHpA | 1.15 J | |
| | PFHxA | 1.47 J | |
| | PFNA | 0.784 J | |
| | PFOA | 0.963 J | |
| | PFPeA | 1.61 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ). Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW5J-2-GW-215-225-01-102621 | L35970-43 | 13C4-PFBA | 16.0 | R |
| EQ-TUBE-110121 | L35970-46 | D5-N-EtFOSA | 49.5 | UJ |
| MW13B-GW-295-305-01-102921 | L35970-48 | D5-N-EtFOSA | 47.5 | UJ |
| MW13D-GW-15-25-01-102921 | L35970-50 | D5-N-EtFOSA | 48.9 | UJ |
| MW14A-GW-316-326-01-102521 | L35970-51 | d9-NEt-FOSE | 46.4 | UJ |
| MW14B-GW-60-70-01-102521 | L35970-52 | D3-MeFOSAA | 48.4 | UJ |
| MW15B-GW-215-225-01-102521 | L35970-55 | 13C4-PFBA | 2.24 | R |
| MW15B-GW-215-225-01-102521 | L35970-55 | 13C5-PFPeA | 32.0 | UJ |
| MW15B-GW-215-225-01-102521 | L35970-55 | 13C2-8:2 FTS | 38.5 | UJ |
| MW15B-GW-215-225-01-102521 | L35970-55 | D3-MeFOSAA | 33.2 | UJ |
| MW15B-GW-215-225-01-102521 | L35970-55 | D5-EtFOSAA | 39.1 | UJ |

**Field Duplicates (Section 6Bi):** The parent sample for field duplicate MW17C-GW-38.5-48.5-02-102621 was reported in WG78957. Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| PFBS | 2.23 | 3.46 | 43 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW5J-1-GW-230-240-01-102621 | L35970-41 | PFOS |
| OW5J-2-GW-215-225-01-102621 | L35970-43 | PFOS |
| MW13B-GW-295-305-01-102921 | L35970-48 | PFOS |
| MW13C-GW-115-125-01-102921 | L35970-49 | PFOS |
| MW13D-GW-15-25-01-102921 | L35970-50 | PFOS |

The PFOS result for sample MW14B-GW-60-70-01-102521 was reported from a dilution analyses due to a high sample concentration.

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# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG79528 Report date (mm/dd/yyyy): 1/4/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/24/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 5.7, 6.3 and 13.7°C, and some of the samples were above the ≤ 6 °C criteria. Results for three samples were qualified as they were received at temperatures > 10 °C.<br>See data qualifications summary (attachment) for exceedances and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | EQ-BLADDER-110121<br>EQ-TUBE-110121<br>EQ-DIPPER-110121<br>Results were nondetect, with the exception of sample EQ-DIPPER-110121.<br>In addition, COCs indicate that TRIP-COOLER10-110321, TRIP-COOLER11-110321, and TRIP-COOLER12-110321 were associated with samples in this SDG. The database indicates the trip blanks were nondetect. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for detections. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | PFAS water limits used: 70% to 130% |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | FDs: MW17C-GW-38.5-48.5-01-102621 / MW17C-GW-38.5-48.5-02-102621 |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>LCS/LCSD -ok<br>FDs - See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications. Two results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/24/2022

**SGS AXYS DPWG79727** 4/24/22 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| EP18E-WAT-BULK-01-111821 | PFOS | 245853 | 20956 | 1 | 2960.40 | 2960 | 0.0638 | 0.004 | L36137-1 |
| EP19-WAT-BULK-01-111821 | PFOS | 86149 | 20204 | 1 | 549.17 | 549 | 0.063 | 0.004 | L36137-2 |
| LCS (WG79331-102) | PFOS | 14664 | 24854 | 1 | 4.75 | 4.75 | 1.00 | 0.004 | WG79331-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 12/17/21 21:47 | 16082 | 28007 | 4.622 | 4.6 | 5.00 | 92.0 | 91.9% |
| CCV 12/18/21 3:13 | 15312 | 25596 | 4.815 | 4.81 | 5.00 | 96.2 | 96.2% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG78095-101): | PFOS | 0.000 | <0.4 | verified <0.4 |

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# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG79727 (Analysis WG79331) dated 1/20/2022 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 4/30/2022 |

The samples listed below were collected at the Project 1007 Site on November 17, 18, and 22 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| EP18E-WAT-BULK-01-111821 | | 11/18/2021 | L36137-1 |
| EP19-WAT-BULK-01-111821 | | 11/18/2021 | L36137-2 |
| EP26A-WAT-BULK-01-111821 | | 11/18/2021 | L36137-3 |
| EP26L-WAT-BULK-01-111821 | | 11/18/2021 | L36137-4 |
| MW10A-GW-236-240-01-111721 | | 11/17/2021 | L36137-5 |
| MW10B-GW-86-90-01-112221 | | 11/22/2021 | L36137-6 |
| MW10B-GW-116-120-01-112221 | | 11/22/2021 | L36137-7 |
| MW10B-GW-116-120-02-112221 | Field Duplicate of MW10B-GW-116-120-01-112221 | 11/22/2021 | L36137-8 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance document listed above, and the analytical method (MLA-110).

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✓ Sample Receipt and Holding times
✓ Initial calibration/continuing calibration verification
✓ Laboratory and trip blanks
✗ Labeled Compounds
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results

✓ Laboratory control sample (LCS)
✓ Field duplicates
✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to labeled compound recoveries, matrix spikes, and quantitations as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at SGS-AXYS intact, correctly preserved, and within the temperature criteria of ≤ 6 °C. Other samples, not reported in this SDG, were included on the chain of custodies.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for a number of analytes (and the two highest standards were excluded for NFDHA) from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification (SSV) recoveries were within ± 30% of the true value.

CCV recoveries were within 100 ± 30% of true known value.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The associated trip blank (TRIP-COOLER1-120121, L36136-1) was reported in data package DPWG79725/ WG79331. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, except as listed in that table below. Results associated with low recoveries were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP18E-WAT-BULK-01-111821 | L36137-1 | D5-N-EtFOSA | 49.0 | UJ |
| EP18E-WAT-BULK-01-111821 | L36137-1 | D3-MeFOSAA | 47.7 | UJ |
| EP18E-WAT-BULK-01-111821 | L36137-1 | d9-NEt-FOSE | 47.4 | UJ |
| EP26A-WAT-BULK-01-111821 | L36137-3 | d7-NMe-FOSE | 47.5 | UJ |
| EP26A-WAT-BULK-01-111821 | L36137-3 | d9-NEt-FOSE | 42.6 | UJ |
| MW10B-GW-86-90-01-112221 | L36137-6 | d9-NEt-FOSE | 47.1 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. The MS/MSD MW10B-GW-116-120-03-112221 associated with samples MW10B-GW-116-120-01-112221 and MW10B-GW-116-120-02-112221 was reported in SDG DPWG79725 (WG79331) and exceedances are summarized below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW10B-GW-116-120-03-112221:** | | | | | |
| PFDoS | 76.6/**68.0** | 70-130 | 14.0 | 30 | The PFDoS result for samples MW10B-GW-116-120-01-112221 and MW10B-GW-116-120-02-112221 were nondetect and qualified as estimated (UJ). |
| PFBA | **-63.9/29.7** | 70-130 | 18.6 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits of 70% to 130% recovery.

**Field Duplicate Results**

Samples MW10B-GW-116-120-01-112221, MW10B-GW-116-120-02-112221, and MW10B-GW-116-120-03-112221 (reported in SDG DPWG79725 (WG79331)) were assess for field precision. RPDs were within the 30 percent limit for water sample, or results were within ± the reporting limit (RL).

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for PFOS and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Reduced sample volume was used for samples EP18E-WAT-BULK-01-111821, EP19-WAT-BULK-01-111821, EP26A-WAT-BULK-01-111821, and EP26L-WAT-BULK-01-111821 due to high analyte concentrations.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| EP26A-WAT-BULK-01-111821 | L36137-3 | PFHxS | No | The PFHxS results were qualified as estimated biased high (J+) as they included quantitation of a branched isomer (PFHxS-II) that did not meet the signal to noise criteria. |
| EP26L-WAT-BULK-01-111821 | L36137-4 | PFHxS | No | |
| MW10A-GW-236-240-01-111721 | L36137-5 | PFHxS<br>PFOS | No | The results were qualified as estimated biased high (J+) as they included quantitation of a branched isomer that did not meet the ion ratio criteria. |
| MW10B-GW-116-120-01-112221 | L36137-7 | PFOA | No | The PFOA result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer (PFOA-II) that did not meet the signal to noise criteria. |
| MW10B-GW-116-120-02-112221 | L36137-8 | PFHxS | No | The result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet the ion ratio criteria. |

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| EP18E-WAT-BULK-01-111821 (L36137-1) | N-EtFOSA<br>MeFOSAA<br>NEt-FOSE | ng/L | UJ | s |
| EP26A-WAT-BULK-01-111821 (L36137-3) | NEt-FOSE<br>NMe-FOSE | ng/L | UJ | s |
| MW10B-GW-86-90-01-112221 (L36137-6) | NEt-FOSE | ng/L | UJ | s |
| MW10B-GW-116-120-01-112221 (L36137-7)<br>MW10B-GW-116-120-02-112221 (L36137-8) | PFDoS | ng/L | UJ | m |
| EP26A-WAT-BULK-01-111821 (L36137-3)<br>EP26L-WAT-BULK-01-111821 (L36137-4)<br>MW10A-GW-236-240-01-111721 (L36137-5)<br>MW10B-GW-116-120-02-112221 (L36137-8) | PFHxS | ng/L | J+ | q |
| MW10A-GW-236-240-01-111721 (L36137-5) | PFOS | ng/L | J+ | q |
| MW10B-GW-116-120-01-112221 (L36137-7) | PFOA | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

m Matrix spike
s Labeled compound
q Quantitation

# Attachment A

**SGS AXYS DPWG79829** 5/1/22 **LS**

Initial Cal 2/22/21 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 31584 | 1588 | 0.4 | 1.2648 | 1.25 |
| 33587 | 3689 | 0.8 | 1.3814 | 1.39 |
| 29945 | 7784 | 2 | 1.3078 | 1.3 |
| 29481 | 17329 | 5 | 1.1829 | 1.17 |
| 29168 | 37350 | 10 | 1.2885 | 1.28 |
| 27901 | 68517 | 20 | 1.2355 | 1.24 |
| 29636 | 178468 | 50 | 1.2119 | 1.20 |
| 26521 | 771756 | 250 | 1.1712 | 1.16 |
| 20023 | 2548643 | 1000 | 1.2807 | 1.26 |
| | | Avg: | 1.2583 | 1.25 |
| | | %RSD: | 5.2577 | 5.50 |

IS Conc = 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| WCLE-GW-0-93-01-110121 | PFOS | 604 | 21803 | 1 | 0.53 | 0.532 | 0.4190 | 0.004 | L36250-2 |
| W20J-3-GW-280-290-01-120221 | PFOS | 9684 | 21706 | 1 | 7.154 | 7.16 | 0.502 | 0.004 | L36250-7 |
| LCS (WG79523-102) | PFOS | 12438 | 21253 | 1 | 4.71 | 4.71 | 1.00 | 0.004 | |
| LCS (WG79523-104) | PFOS | 12771 | 21185 | 1 | 4.85 | 4.85 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 1/15/22 3:33 | 20404 | 34083 | 4.819 | 4.82 | 5.00 | 96.4 | 96.4% |
| CCV 1/15/22 10:14 | 20713 | 35007 | 4.763 | 4.76 | 5.00 | 95.3 | 95.3% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG79523-101): | PFOS | 0.000 | <0.5 | verified <0.5 |

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# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG79829 (Analysis WG79523) dated 1/27/2022 |
| Analyses/Method: | SGS AXYS Method MLA-110 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 4/30/2022 |

The samples listed below were collected at the Project 1007 Site on December 2 and 8, 2021.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| OW20S-1-GW-190-200-01-120221 | | 12/2/2021 | L36250-2 |
| MW10A-GW-273-283-01-120821 | | 12/8/2021 | L36250-3 |
| MW10B-GW-130-140-01-120221 | | 12/2/2021 | L36250-4 |
| PW20J-1-GW-308-358-01-120221 | | 12/2/2021 | L36250-5 |
| OW20J-1-GW-310-320-01-120221 | | 12/2/2021 | L36250-6 |
| OW20J-3-GW-280-290-01-120221 | | 12/2/2021 | L36250-7 |
| **Field QC Blanks:** | | | |
| TRIP-COOLER1-122021 | Trip Blank | 12/20/2021 | L36250-1 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance document listed above, and the analytical method (MLA-110).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✗ | Initial calibration/continuing calibration verification |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control sample (LCS) |

| | |
|---|---|
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to calibration verification, labeled compound recoveries, laboratory control samples, and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at SGS-AXYS intact, correctly preserved, and within the temperature criteria of ≤ 6 °C.

The field sample ID for sample L36250-1 was updated from TRIPBLANK-COOLER1-122021 to TRIP-COOLER1-122021.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

### Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for a number of analytes (and the two highest standards were excluded for NFDHA) from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification (SSV) recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency, CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/15/22 3:33 | ADONA | 132% | 70-130% | Associated results were nondetect. No qualifiers. |
| | NFDHA | 26.2% | 70-130% | NFDHA results were qualified as not quantifiable (NQ) by the laboratory, and no result was reported. Results were rejected (R) in the database as discussed in the Quantitation and Results Verification section. |
| CCV 1/15/22 10:14 | 4:2 FTS | 131% | 70-130% | Associated results were nondetect. No qualifiers. |
| | 7:3 FTCA | 66.6% | 70-130% | Associated results were nondetect and qualified as estimated (UJ). |
| | NFDHA | 40.5% | 70-130% | NFDHA results were qualified as not quantifiable (NQ) by the laboratory, and no result was reported. Results were rejected (R) in the database as discussed in the Quantitation and Results Verification section. |

Two CCVs were associated with this data set and results were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank was nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blank (TRIP-COOLER1-122021) blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, except as listed in that table below. Results associated with low recoveries were qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-122021 | L36250-1 | d9-NEt-FOSE | 42.4 | UJ |
| OW20S-1-GW-190-200-01-120221 | L36250-2 | D5-N-EtFOSA | 48.9 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | D3-N-MeFOSA | 46.8 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | D5-N-EtFOSA | 40.2 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | D3-MeFOSAA | 42.9 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | D5-EtFOSAA | 49.2 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | d7-NMe-FOSE | 42.8 | UJ |
| MW10A-GW-273-283-01-120821 | L36250-3 | d9-NEt-FOSE | 32.5 | UJ |
| MW10B-GW-130-140-01-120221 | L36250-4 | D5-N-EtFOSA | 45.1 | UJ |
| MW10B-GW-130-140-01-120221 | L36250-4 | D3-MeFOSAA | 45.3 | UJ |
| MW10B-GW-130-140-01-120221 | L36250-4 | D5-EtFOSAA | 47.2 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW10B-GW-130-140-01-120221 | L36250-4 | d7-NMe-FOSE | 46.1 | UJ |
| MW10B-GW-130-140-01-120221 | L36250-4 | d9-NEt-FOSE | 34.3 | UJ |
| PW20J-1-GW-308-358-01-120221 | L36250-5 | d9-NEt-FOSE | 44.5 | UJ |
| OW20J-1-GW-310-320-01-120221 | L36250-6 | d9-NEt-FOSE | 46.7 | UJ |
| OW20J-3-GW-280-290-01-120221 | L36250-7 | D3-MeFOSAA | 45.2 | UJ |
| OW20J-3-GW-280-290-01-120221 | L36250-7 | D5-EtFOSAA | 48.3 | UJ |
| OW20J-3-GW-280-290-01-120221 | L36250-7 | d7-NMe-FOSE | 47.4 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSDs were not associated with this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits except as listed in the table below.

| LCS/LCSD IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG79523-102 WG79523-104 | NFDHA | **42.9/27.7** | 70-130 | NFDHA results were qualified as not quantifiable (NQ) by the laboratory, and no result was reported. Results were rejected (R) in the database as discussed in the Quantitation and Results Verification section. |
| | 9Cl-PF3ONS | 114/**141** | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | ADONA | 127/**144** | 70-130 | |

**Field Duplicate Results**

Field duplicates are collected to assess both laboratory and field precision. Field duplicates were not associated with this data set.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for PFOS and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below. Qualifications are discussed in the table below.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum | Reason |
|---|---|---|---|---|
| OW20S-1-GW-190-200-01-120221 | L36250-2 | PFHxA<br>PFOS | No | The results were qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet signal to noise criteria. |
| MW10A-GW-273-283-01-120821 | L36250-3 | PFOA | No | |
| MW10A-GW-273-283-01-120821 | L36250-3 | PFOS | Yes | The result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet the ion ratio and signal to noise criteria. |
| MW10B-GW-130-140-01-120221 | L36250-4 | PFOA<br>PFHxS | No | The results were qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet signal to noise criteria. |
| MW10B-GW-130-140-01-120221 | L36250-4 | PFOS | Yes | The result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet the ion ratio and signal to noise criteria. |
| PW20J-1-GW-308-358-01-120221 | L36250-5 | PFOA | No | The result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet signal to noise criteria. |
| OW20J-3-GW-280-290-01-120221 | L36250-7 | PFOS | Yes | The result was qualified as estimated biased high (J+) as it included quantitation of a branched isomer that did not meet the ion ratio criteria. |

NFDHA results were qualified as not quantifiable (NQ) by the laboratory, as this compound did not adequately meet several method performance indicators. A value was not reported in the laboratory report and these results were rejected (R) in the database.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All samples | 7:3 FTCA | ng/L | UJ | c |
| TRIP-COOLER1-122021 (L36250-1)<br>PW20J-1-GW-308-358-01-120221 (L36250-5)<br>OW20J-1-GW-310-320-01-120221 (L36250-6) | NEt-FOSE | ng/L | UJ | s |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| OW20S-1-GW-190-200-01-120221 (L36250-2) | N-EtFOSA | ng/L | UJ | s |
| MW10A-GW-273-283-01-120821 (L36250-3) | N-MeFOSA<br>N-EtFOSA<br>MeFOSAA<br>EtFOSAA<br>NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| MW10B-GW-130-140-01-120221 (L36250-4) | N-EtFOSA<br>MeFOSAA<br>EtFOSAA<br>NMe-FOSE<br>NEt-FOSE | ng/L | UJ | s |
| OW20J-3-GW-280-290-01-120221 (L36250-7) | NEt-FOSE<br>EtFOSAA<br>NMe-FOSE | ng/L | UJ | s |
| MW10A-GW-273-283-01-120821 (L36250-3)<br>MW10B-GW-130-140-01-120221 (L36250-4)<br>OW20J-3-GW-280-290-01-120221 (L36250-7) | PFOS | ng/L | J+ | em |
| OW20S-1-GW-190-200-01-120221 (L36250-2) | PFHxA<br>PFOS | ng/L | J+ | q |
| MW10A-GW-273-283-01-120821 (L36250-3) | PFOA | ng/L | J+ | q |
| MW10B-GW-130-140-01-120221 (L36250-4) | PFOA<br>PFHxS | ng/L | J+ | q |
| PW20J-1-GW-308-358-01-120221 (L36250-5) | PFOA | ng/L | J+ | q |
| All samples | NFDHA | ng/L | R | c,l,q |

(1): Data Validation Qualifiers:

- J Estimated (+/- indicates high or low bias)
- R Rejected
- UJ Estimated reporting limit

(2): Reason Codes:

- c Calibration
- em Estimated maximum concentration
- l Laboratory control sample
- q Quantitation
- s Labeled compound

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG80441 (WG80181)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 3/9/22 18:06 | NFDHA | 46.9% | 70-130% | All NFDHA results were associated with these CCVs and were qualified UJ. |
| CCV 3/10/22 00:42 | NFDHA | 44.9% | 70-130% | |
| CCV 3/11/22 12:37 | NFDHA | 60.8% | 70-130% | NFDHA results were not associated with this CCV. |
| CCV 3/11/22 13:43 | N-EtFOSA | 66.5% | 70-130% | N-EtFOSA results were not associated with this CCV. No qualifiers. |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and the results qualified are listed below.

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG80181-102 | NFDHA | **57.9/67.3** | 70-130 | 14.9 | 30 | Associated results were nondetect and qualified UJ. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG80441 Report date (mm/dd/yyyy): 3/17/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/9/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☐ | ☒ | ☐ | Volume for an MS/MSD was not included in this SDG; however, MS/MSDs were collected for this sampling event as shown on the COCs. |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-020922 (L36495-1)<br>TRIP-COOLER2-020922 (L36495-2)<br>TRIP-COOLER3-020922 (L36495-3)<br>TRIP-COOLER4-020922 (L36495-4)<br>TRIP-COOLER5-020922 (L36495-5)<br>TRIP-COOLER6-020922 (L36495-6) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | TRIP-COOLER1-020922 (L36495-1):<br>6:2 FTS 11.1 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | Sample results were nondetect. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☒ | ☐ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | PZDS-GW-20-30-01-020322 / PZDS-GW-20-30-02-020322 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix<br>RPDs for FD - ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>Reduced sample volumes were used for six samples.<br>PFOS was reported from dilution analyses for samples PZBD-GW-16-21-01-020222 and PZFS-GW-18-28-01-012722 ( L36495-10 and -18) due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/8/2022

**SGS AXYS DPWG79727** 6/28/22 **LS**

Initial Cal 2/14/22 **PFOS**

| Area Counts | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | | | | |
| | | | | | | | | |
| 34265 | 1593 | 0.4 | 1.1695 | 1.16 | | | | |
| 23013 | 2316 | 0.8 | 1.2658 | 1.27 | | | | |
| 22120 | 5369 | 2 | 1.2211 | 1.22 | | | | |
| 20440 | 11954 | 5 | 1.1769 | 1.17 | | | | |
| 23544 | 26358 | 10 | 1.1265 | 1.12 | | | | |
| 22751 | 53583 | 20 | 1.1849 | 1.18 | | | | |
| 21793 | 131966 | 50 | 1.2186 | 1.21 | | | | |
| 19333 | 581226 | 250 | 1.2100 | 1.20 | | | | |
| | | Avg: | 1.1967 | 1.19 | 1.19184 | (raw data) | | |
| | | %RSD: | 3.4932 | 3.61 | | Internal Std Conc: | 10.062 | |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW18B-GW-225-235-01-013122 | PFOS | 14492 | 23960 | 1 | 10.53 | 10.5 | 0.4850 | 0.004 | L36505-2 |
| OW20J-1-GW-310-320-01- | PFOS | 82354 | 20355 | 1 | 65.81 | 65.8 | 0.519 | 0.004 | L36505-5 |
| LCS ( WG80185-102) | PFOS | 12300 | 20223 | 1 | 5.13 | 5.09 | 1.00 | 0.004 | WG80185-102 |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 3/4/22 16:03 | 19007 | 33680 | 4.764 | 4.76 | 5.00 | 95.2 | 95.3% |
| CCV 3/4/22 18:41 | 16300 | 27558 | 4.994 | 5.12 | 5.00 | 102.4 | 102% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG80185-101): | PFOS | 0.027 | <0.4 | verified <0.4 |

AECOM
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Milwaukee, WI 53212
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414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MPCA – Project 1007 |
| Laboratory: | SGS AXYS – Sidney, BC, Canada |
| Data Package: | DPWG80520 (Analysis WG80185) dated 3/22/2022 |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 |
| Validation Level: | Stage 4 |
| Prepared by: | Lisa Smith (CEAC) Completed on: 6/29/2022 |

The samples listed below were collected at the Project 1007 Site on January 12, 13, 31, and February 9, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| MW18B-GW-225-235-01-013122 | | 1/31/2022 | L36505-2 |
| MW18B-GW-116-120-01-011222 | | 1/12/2022 | L36505-3 |
| MW18B-GW-176-180-01-011322 | | 1/13/2022 | L36505-4 |
| OW20J-1-GW-310-320-01-013122 | | 1/31/2022 | L36505-5 |
| OW20J-2-GW-310-320-01-013122 | | 1/31/2022 | L36505-6 |
| OW20J-3-GW-280-290-01-013122 | | 1/31/2022 | L36505-7 |
| OW20S-1-GW-190-200-01-013122 | | 1/31/2022 | L36505-8 |
| **Field QC Samples:** | | | |
| TRIP-COOLER7-020922 | Trip Blank | 2/9/2022 | L36505-1 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✓ Sample Receipt and Holding times
✗ Initial calibration/continuing calibration verification
✓ Laboratory and trip blanks
✗ Labeled Compounds

| | |
|---|---|
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control samples (LCSs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to continuing calibration verification, labeled compound recoveries, LCSs, and quantitations (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at SGS-AXYS intact, correctly preserved, and within the temperature criteria of ≤ 6 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

### Calibration

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for NFDHA from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 67.7% for the highest calibration point. NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification (SSV) recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 3/4/22 16:03 | NFDHA | 67.8% | 70-130% | NFDHA results were nondetect and qualified as estimated (UJ). |
| CCV 3/4/22 18:41 | NFDHA | 68.0% | 70-130% | |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW18B-GW-116-120-01-011222 | L36505-3 | D5-EtFOSAA | 44.6 | UJ |
| OW20S-1-GW-190-200-01-013122 | L36505-8 | 13C4-PFBA | 46.6 | J+ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits except as listed in the table below.

| LCS IDs | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG80185-102 | NFDHA | **61.2** | 70-130 | The associated results were nondetect and qualified UJ. |
| WG80185-103 | N-MeFOSE | **66.6** | 70-130 | The associated results were nondetect and qualified UJ. |
| | NFDHA | **63.9** | 70-130 | |

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results reported as listed in the table below which did not meet ion ratio criteria and were qualified as estimated biased high (J+).

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW18B-GW-225-235-01-013122 | L36505-2 | PFHxS | No |
| | | PFOS | No |
| MW18B-GW-116-120-01-011222 | L36505-3 | PFOS | No |
| MW18B-GW-176-180-01-011322 | L36505-4 | PFOS | No |
| OW20J-1-GW-310-320-01-013122 | L36505-5 | PFOS | No |
| OW20J-2-GW-310-320-01-013122 | L36505-6 | PFNA | Yes |
| | | PFOS | No |
| OW20J-3-GW-280-290-01-013122 | L36505-7 | PFHxS | No |
| OW20S-1-GW-190-200-01-013122 | L36505-8 | PFOS | Yes |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW18B-GW-116-120-01-011222 (L36505-3) | EtFOSAA | ng/L | UJ | s |
| OW20S-1-GW-190-200-01-013122 (L36505-8) | PFBA | ng/L | J+ | s |
| All samples | N-MeFOSE | ng/L | UJ | l |
| All samples | NFDHA | ng/L | UJ | c,l |
| MW18B-GW-225-235-01-013122 (L36505-2) | PFHxS | ng/L | J+ | q |
| | PFOS | ng/L | J+ | q |
| MW18B-GW-116-120-01-011222 (L36505-3) | PFOS | ng/L | J+ | q |
| MW18B-GW-176-180-01-011322 (L36505-4) | PFOS | ng/L | J+ | q |
| OW20J-1-GW-310-320-01-013122 (L36505-5) | PFOS | ng/L | J+ | q |
| OW20J-2-GW-310-320-01-013122 (L36505-6) | PFNA | ng/L | J+ | em |
| | PFOS | ng/L | J+ | q |
| OW20J-3-GW-280-290-01-013122 (L36505-7) | PFHxS | ng/L | J+ | q |
| OW20S-1-GW-190-200-01-013122 (L36505-8) | PFOS | ng/L | J+ | em |

(1): Data Validation Qualifiers:

| | |
|---|---|
| J | Estimated (+/- indicates high or low bias) |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| c | Continuing calibration verification |
| em | Estimated maximum concentration |
| l | Laboratory control sample |
| s | Labeled compound |
| q | Quantitation |

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG81221 (WG80849)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/5/22 5:33 | NFDHA | 63.1% | 70-130% | NFDHA results associated with this CCV were nondetect and qualified as estimated (UJ):<br><br>MW2B-GW-57-62-02-032922 (L36841-22)<br>MW2B-GW-57-62-03-032922 (L36841-23 (A))<br>MW14C-GW-16-36-01-032922 (L36841-27)<br>MW14D-GW-6-21-01-032922 (L36841-26) |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW2B-GW-57-62-03-032922:** | | | | | |
| PFOA | **148/57.6** | 70-130 | 2.96 | 30 | The sample concentrations were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **804/849** | 70-130 | 0.551 | 30 | |
| NFDHA | 70.5/122 | 70-130 | **39.4** | 30 | The result for sample MW2B-GW-57-62-03-032922 was nondetect, and was acceptable without qualification. |

**Bold indicates an exceedance**

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81221 (WG80849) Report date (mm/dd/yyyy): 5/20/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/7/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | CoCs indicate the trip blanks listed below were associated with the samples (they were not reported in this SDG):<br>TRIP-COOLER2-040622 (L36841-2)<br>TRIP-COOLER3-040622 (L36841-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2B-GW-57-62-03-032922 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary for exceedances. Qualifications not required. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Triplicate: MW2B-GW-57-62-01-032922 / MW2B-GW-57-62-02-032922 / MW2B-GW-57-62-03-032922<br>Field Duplicate: OD5-WAT-BULK-01-032822 / OD5-WAT-BULK-02-032822 (parent sample reported in WG80850). |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | MSD was submitted for MW2B-GW-57-62-03-032922 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix<br>RPDs for field triplicate/duplicate - ok<br>MS/MSD RPD for NFDHA was above 30%. The sample result was nondetect. No qualifiers. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>A reduced sample volume was used for sample MW14B-GW-60-70-01-032922 (L36841-24).<br>PFOS dilutions were required due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/7/2022

**SGS AXYS DPWG79727** 8/15/22 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | | | | | |
|---|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | | p 773 |
| | | | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 | | |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 | | |
| 27916 | 6263 | 2 | 1.1287 | 1.13 | | |
| 23878 | 13307 | 5 | 1.1215 | 1.11 | | |
| 24247 | 28296 | 10 | 1.1742 | 1.17 | | |
| 26274 | 63187 | 20 | 1.2099 | 1.21 | | |
| 26621 | 144547 | 50 | 1.0927 | 1.09 | | |
| 25106 | 683620 | 250 | 1.0959 | 1.09 | | |
| | | Avg: | 1.1373 | 1.13 | 1.13401 | (raw data) |
| | | %RSD: | 5.9989 | 6.15 | | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| EP27I-WAT-BULK-01-042122 | PFOS | 492945 | 21458 | 1 | 382.43 | 383 | 0.5330 | 0.004 | L36997-41 |
| EP18E-WADE-WAT-BULK-01- | PFOS | 408908 | 18971 | 1 | 400.11 | 400 | 0.478 | 0.004 | L36997-46 |
| LCS WG81198-10 | PFOS | 11374 | 21616 | 1 | 4.67 | 4.67 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/2/22 19:14 | 16504 | 30966 | 4.729 | 4.7 | 5.00 | 94.0 | 94.0% |
| CCV 6/2/22 22:42 | 16908 | 31760 | 4.724 | 4.71 | 5.00 | 94.2 | 94.2% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG81198-101): | PFOS | 0.046 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG81516 (Analysis WG81198) dated 6/15/2022 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 8/26/2022 |

The samples listed below were collected at the Project 1007 Site on April 18 and 21, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| EP27I-WAT-BULK-01-042122 | | 4/21/2022 | L36997-41 |
| EP27I-WADE-WAT-BULK-01-042122 | | 4/21/2022 | L36997-42 |
| EP2A-WAT-BULK-01-042122 | | 4/21/2022 | L36997-43 |
| EP2A-WADE-WAT-BULK-01-042122 | | 4/21/2022 | L36997-44 |
| EP18E-WAT-BULK-01-042122 | | 4/21/2022 | L36997-45 |
| EP18E-WADE-WAT-BULK-01-042122 | | 4/21/2022 | L36997-46 |
| EP26D-WAT-BULK-01-042122 | | 4/21/2022 | L36997-47 |
| EP26D-WADE-WAT-BULK-01-042122 | | 4/21/2022 | L36997-48 |
| EP19-WAT-BULK-01-042122 | | 4/21/2022 | L36997-49 |
| EP26A-WAT-BULK-01-042122 | | 4/21/2022 | L36997-50 |
| EP27A-WAT-BULK-01-042122 | | 4/21/2022 | L36997-51 |
| EP24-WAT-BULK-01-041822 | | 4/18/2022 | L36997-52 |
| EP8A-WAT-BULK-01-042122 | | 4/21/2022 | L36997-53 |
| WL6-WAT-BULK-01-041822 | | 4/18/2022 | L36997-54 |
| WL17A-WAT-BULK-01-041822 | | 4/18/2022 | L36997-55 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✗ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control samples/laboratory control sample duplicates (LCS/LCSDs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Twelve PFOSA results were qualified BJ/B due to trip contamination. In addition, results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to labeled compound recoveries and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact. The samples were received above 6 °C at temperatures of 6.6 and 8.6 °C. Results were not qualified as the samples were received at temperatures ≤ 10 °C.

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA, and PFUnA from the initial calibration curve due to non-linearity at the high standard concentration.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point. NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification (SSV) recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/2/22 22:04 | N-MeFOSE | 142% | 70-130% | N-MeFOSE results were nondetect and were acceptable without qualification. |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. The trip blanks associated with this sample set (TRIP-COOLER6-050422 and TRIP-COOLER6-050422) were reported in SDG DPWG81551 (WG81196) and were nondetect, with the exception of TRIP-COOLER6-050422. PFOSA was detected in the trip blank at a concentration of 0.512 J ng/L. Associated sample detects within 10 times the blank concentration were qualified B/BJ.

## Labeled Compounds

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP27I-WAT-BULK-01-042122 | L36997-41 | 13C4-PFBA | 18.5 | J+ |
| EP27I-WADE-WAT-BULK-01-042122 | L36997-42 | 13C4-PFBA | 22.6 | J+ |
| EP2A-WADE-WAT-BULK-01-042122 | L36997-44 | 13C4-PFBA | 20.9 | J+ |
| EP18E-WAT-BULK-01-042122 | L36997-45 | 13C2-PFTeDA | 44.9 | UJ<br>PFTrDA: UJ |
| EP18E-WAT-BULK-01-042122 | L36997-45 | D3-N-MeFOSA | 41.9 | UJ |
| EP26D-WADE-WAT-BULK-01-042122 | L36997-48 | 13C4-PFBA | 45.4 | J+ |
| EP19-WAT-BULK-01-042122 | L36997-49 | 13C4-PFBA | 13.7 | J+ |
| EP24-WAT-BULK-01-041822 | L36997-52 | 13C4-PFBA | 27.9 | J+ |
| EP8A-WAT-BULK-01-042122 | L36997-53 | 13C4-PFBA | 35.4 | J+ |
| WL6-WAT-BULK-01-041822 | L36997-54 | 13C4-PFBA | 47.9 | J+ |
| WL17A-WAT-BULK-01-041822 | L36997-55 | 13C4-PFBA | 42.4 | J+ |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

## LCS/LCSD Results

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits, except as listed in the table below.

| LCS IDs | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81198-103 (Low Range) | N-EtFOSA | 62.4 | 70-130 | The associated results were nondetect and qualified UJ. |
| | NFDHA | 163 | 70-130 | NFDHA results were nondetect and were acceptable without qualification. |

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

The PFOS result for sample EP26D-WAT-BULK-01-042122 was reported from a dilution analysis due to the high sample concentration.

A smaller sample volume was used for sample prep for EP2A-WAT-BULK-01-042122 due to the high solids content. In addition, a smaller sample volume was used for EP18E-WAT-BULK-01-042122 due to a high PFOS concentration. The smaller sample volumes cause high reporting limits.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and were qualified as estimated biased high (J+).

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| EP27I-WAT-BULK-01-042122 | L36997-41 | PFOS | No |
| EP27I-WADE-WAT-BULK-01-042122 | L36997-42 | PFOS | No |
| EP2A-WAT-BULK-01-042122 | L36997-43 | PFBS | Yes |
| EP2A-WAT-BULK-01-042122 | L36997-43 | PFHxS | No |
| EP2A-WAT-BULK-01-042122 | L36997-43 | PFOS | No |
| EP2A-WADE-WAT-BULK-01-042122 | L36997-44 | PFOS | No |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| EP18E-WAT-BULK-01-042122 | L36997-45 | PFOS | No |
| EP18E-WAT-BULK-01-042122 | L36997-45 | EtFOSAA | No |
| EP18E-WADE-WAT-BULK-01-042122 | L36997-46 | PFHxS | No |
| EP18E-WADE-WAT-BULK-01-042122 | L36997-46 | PFOS | No |
| EP18E-WADE-WAT-BULK-01-042122 | L36997-46 | EtFOSAA | No |
| EP26D-WAT-BULK-01-042122 | L36997-47 | PFOS | No |
| EP26D-WADE-WAT-BULK-01-042122 | L36997-48 | PFOS | No |
| EP19-WAT-BULK-01-042122 | L36997-49 | PFOS | No |
| EP26A-WAT-BULK-01-042122 | L36997-50 | PFHxS | No |
| EP26A-WAT-BULK-01-042122 | L36997-50 | PFOS | No |
| EP27A-WAT-BULK-01-042122 | L36997-51 | PFOS | No |
| EP24-WAT-BULK-01-041822 | L36997-52 | PFOS | No |
| EP8A-WAT-BULK-01-042122 | L36997-53 | PFOS | No |
| EP8A-WAT-BULK-01-042122 | L36997-53 | EtFOSAA | No |
| WL6-WAT-BULK-01-041822 | L36997-54 | PFOS | No |
| WL17A-WAT-BULK-01-041822 | L36997-55 | PFOS | No |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| EP18E-WAT-BULK-01-042122 (L36997-45) | PFTeDA<br>PFTrDA<br>N-MeFOSA | ng/L | UJ | s |
| EP27I-WAT-BULK-01-042122 (L36997-41)<br>EP27I-WADE-WAT-BULK-01-042122 (L36997-42)<br>EP2A-WADE-WAT-BULK-01-042122 (L36997-44)<br>EP26D-WADE-WAT-BULK-01-042122 (L36997-48)<br>EP19-WAT-BULK-01-042122 (L36997-49)<br>EP24-WAT-BULK-01-041822 (L36997-52)<br>EP8A-WAT-BULK-01-042122 (L36997-53)<br>WL6-WAT-BULK-01-041822 (L36997-54)<br>WL17A-WAT-BULK-01-041822 (L36997-55) | PFBA | ng/L | J+ | s |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| EP27I-WAT-BULK-01-042122 (L36997-41)<br>EP27I-WADE-WAT-BULK-01-042122 (L36997-42)<br>EP2A-WAT-BULK-01-042122 (L36997-43)<br>EP2A-WADE-WAT-BULK-01-042122 (L36997-44)<br>EP18E-WAT-BULK-01-042122 (L36997-45)<br>EP18E-WADE-WAT-BULK-01-042122 (L36997-46)<br>EP26D-WAT-BULK-01-042122 (L36997-47)<br>EP26D-WADE-WAT-BULK-01-042122 (L36997-48)<br>EP19-WAT-BULK-01-042122 (L36997-49)<br>EP26A-WAT-BULK-01-042122 (L36997-50)<br>EP27A-WAT-BULK-01-042122 (L36997-51)<br>EP8A-WAT-BULK-01-042122 (L36997-53) | PFOSA | ng/L | B/BJ | tb |
| All Samples | N-EtFOSA | ng/L | UJ | l |
| EP18E-WAT-BULK-01-042122 (L36997-45)<br>EP18E-WADE-WAT-BULK-01-042122 (L36997-46)<br>EP8A-WAT-BULK-01-042122 (L36997-53) | EtFOSAA | ng/L | J+ | q |
| EP2A-WAT-BULK-01-042122 (L36997-43)<br>EP18E-WADE-WAT-BULK-01-042122 (L36997-46)<br>EP26A-WAT-BULK-01-042122 (L36997-50) | PFHxS | ng/L | J+ | q |
| All Samples | PFOS | ng/L | J+ | q |
| EP2A-WAT-BULK-01-042122 (L36997-43) | PFBS | ng/L | J+ | em |

(1): Data Validation Qualifiers:

- B Blank contamination
- J Estimated (+/- indicates high or low bias)
- UJ Estimated reporting limit

(2): Reason Codes:

- em Estimated maximum concentration
- l Laboratory control sample
- q Quantitation
- s Labeled compound
- tb Trip blank

**Attachment A**

**SGS AXYS DPWG79727** 9/23/22 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | | | | | |
|---|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | | p 895 |
| | | | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 | | |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 | | |
| 27916 | 6263 | 2 | 1.1287 | 1.13 | | |
| 23878 | 13307 | 5 | 1.1215 | 1.11 | | |
| 24247 | 28296 | 10 | 1.1742 | 1.17 | | |
| 26274 | 63187 | 20 | 1.2099 | 1.21 | | |
| 26621 | 144547 | 50 | 1.0927 | 1.09 | | |
| 25106 | 683620 | 250 | 1.0959 | 1.09 | | |
| | | Avg: | 1.1373 | 1.13 | 1.13401 | (raw data) |
| | | %RSD: | 5.9989 | 6.15 | | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| PZAD-GW-21-26-01-041922 | PFOS | 6852 | 20935 | 1 | 5.83 | 5.83 | 0.498 | 0.004 | L36997-7 |
| MW1B-GW-100-120-01-041822 | PFOS | 10203 | 20175 | 1 | 71.91 | 71.9 | 0.0624 | 0.004 | L36997-20 |
| WG81196-102 | PFOS | 13765 | 24320 | 1 | 5.02 | 5.02 | 1.00 | 0.004 | |
| WG81196-103 | PFOS | 3791 | 23191 | 1 | 1.45 | 1.45 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/6/22 18:20 | 14927 | 27154 | 4.878 | 4.88 | 5.00 | 97.6 | 97.6% |
| CCV 6/7/22 1:57 | 15630 | 28774 | 4.820 | 4.82 | 5.00 | 96.4 | 96.4% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG81196-101): | PFOS | 0.041 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG81551 (Analysis WG81196) dated 7/21/2022 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 9/24/2022 |

The samples listed below were collected at the Project 1007 Site on April 18 through 21, April 25, and May 4, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| PZAS-GW-6-16-01-041922 | | 4/19/2022 | L36997-6 |
| PZAD-GW-21-26-01-041922 | | 4/19/2022 | L36997-7 |
| PZGS-GW-20-30-01-041922 | | 4/19/2022 | L36997-8 |
| PZGD-GW-35-40-01-041922 | Field Duplicate reported in: DPWG81606 (WG81251) | 4/19/2022 | L36997-9 |
| PZHS-GW-6-16-01-041922 | | 4/19/2022 | L36997-10 |
| PZHD-GW-21-26-01-041922 | | 4/19/2022 | L36997-11 |
| PZIS-GW-7-17-01-041922 | | 4/19/2022 | L36997-12 |
| PZID-GW-22-27-01-041922 | | 4/19/2022 | L36997-13 |
| OW20J-1-GW-310-320-01-042522 | | 4/25/2022 | L36997-14 |
| OW20S-1-GW-190-200-01-042522 | | 4/25/2022 | L36997-15 |
| PL41-GW-72-82-01-041922 | | 4/19/2022 | L36997-16 N |
| SP42-GW-116-126-01-041922 | | 4/19/2022 | L36997-17 |
| W6102-GW-93-105-01-042122 | | 4/21/2022 | L36997-18 |
| W6201-GW-109-124-01-041922 | | 4/19/2022 | L36997-19 |
| MW1B-GW-100-120-01-041822 | | 4/18/2022 | L36997-20 |
| **Field QC Samples:** | | | |
| TRIP-COOLER3-050422 | Trip Blank | 5/4/2022 | L36997-1 |
| TRIP-COOLER4-050422 | Trip Blank | 5/4/2022 | L36997-2 |
| TRIP-COOLER5-050422 | Trip Blank | 5/4/2022 | L36997-3 |
| TRIP-COOLER6-050422 | Trip Blank | 5/4/2022 | L36997-4 |
| TRIP-COOLER7-050422 | Trip Blank | 5/4/2022 | L36997-5 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.

- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification |
| ✗ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control samples/laboratory control sample duplicates (LCS/LCSDs) |
| ✗ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of one result that was rejected (R) due to a severely low labeled compound recovery. Three PFOSA results were qualified BJ/B due to trip contamination. In addition, results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to labeled compound recoveries, LCS recoveries, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact. The samples were received above 6 °C at temperatures ranging from 6.6 to 9.2 °C. Results were not qualified as the samples were received at temperatures ≤ 10 °C.

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

## Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA (highest 2 standards), and PFUnA from the initial calibration curve due to non-linearity at the high concentrations.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point, and this calibration point was not used during calibration. In addition, the NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/7/22 1:57 | NFDHA | 139 | 70-130 | NFDHA results were nondetect and were acceptable without qualification. |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Trip blank detections are summarized in the table below. Associated sample detects within 10 times the blank concentration were qualified B/BJ.

| Trip Blank | Analyte | Concentration (ng/L) | Qualifiers |
|---|---|---|---|
| TRIP-COOLER3-050422 | PFOSA | 0.426 J | Associated sample detects were qualified BJ:<br><br>PZAS-GW-6-16-01-041922<br>PZAD-GW-21-26-01-041922<br>PZHD-GW-21-26-01-041922 |
| TRIP-COOLER5-050422 | PFOSA | 0.498 J | Associated samples were not reported in this SDG. |

| Trip Blank | Analyte | Concentration (ng/L) | Qualifiers |
|---|---|---|---|
| TRIP-COOLER6-050422 | PFOSA | 0.512 J | Associated samples were not reported in this SDG. |

## Labeled Compounds

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER3-050422 | L36997-1 | d9-NEt-FOSE | 45.1 | UJ |
| PZAS-GW-6-16-01-041922 | L36997-6 | d9-NEt-FOSE | 47.1 | UJ |
| PZAS-GW-6-16-01-041922 | L36997-6 | d7-NMe-FOSE | 47.2 | UJ |
| PZHS-GW-6-16-01-041922 | L36997-10 | d9-NEt-FOSE | 41.1 | UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | 13C2-PFDoA | 46.9 | UJ<br>PFTrDA: UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | 13C2-PFTeDA | 19.9 | UJ<br>PFTrDA: UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | D3-N-MeFOSA | 38.6 | UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | D5-N-EtFOSA | 37.3 | UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | d7-NMe-FOSE | 20.7 | UJ |
| PZID-GW-22-27-01-041922 | L36997-13 | d9-NEt-FOSE | 9.66 | R |
| OW20S-1-GW-190-200-01-042522 | L36997-15 | D3-N-MeFOSA | 48.5 | UJ |
| OW20S-1-GW-190-200-01-042522 | L36997-15 | d9-NEt-FOSE | 44.5 | UJ |
| W6102-GW-93-105-01-042122 | L36997-18 | 13C4-PFBA | 30.3 | J+ |
| MW1B-GW-100-120-01-041822 | L36997-20 | D3-N-MeFOSA | 48.9 | UJ |
| MW1B-GW-100-120-01-041822 | L36997-20 | d7-NMe-FOSE | 46.7 | UJ |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

## LCS/LCSD Results

LCS/LCSDs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits, except as listed in the table below.

| LCS IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | LCS RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG81197-103 / WG81196-105 | NFDHA | 88.5/**135** | 70-130 | **41.6** | 30 | NFDHA results were nondetect and were acceptable without qualification. |
| WG81196-103 (Low Level) | N-MeFOSA | **65.4/--** | 70-130 | -- | -- | Associated results were nondetect and qualified as estimated (UJ). |

Bold indicates an exceedance

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. The field duplicate for sample PZGD-GW-35-40-01-041922 (L36997-9) was reported DPWG81606 (WG81251). RPDs were within the 30 percent limit for water sample, or results were within ± the reporting limit (RL).

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

The PFBA result for sample PL41-GW-72-82-01-041922 was reported from a dilution analysis due to the high sample concentration.

A smaller sample volume was used for sample prep for samples OW20S-1-GW-190-200-01-042522, SP42-GW-116-126-01-041922 W6201-GW-109-124-01-041922, and MW1B-GW-100-120-01-041822 due to the high particulate content. In addition, the case narrative indicates that some of the sample did not pass through the SPE cartridge for samples PZAS-GW-6-16-01-041922 and PL41-GW-72-82-01-041922, and that a sample size error in the raw data was corrected for these two samples and is documented on the Audit Change Reports.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and were qualified as estimated biased high (J+). PFOS qualifications are mostly attributed to ion ratio exceedances of PFOS-IV which is present at a very low concentration, but does not meet ion ratio criteria.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| PZAS-GW-6-16-01-041922 | L36997-6 | PFHxS | No |
| PZAD-GW-21-26-01-041922 | L36997-7 | PFOS | No |
| PZGS-GW-20-30-01-041922 | L36997-8 | PFOS | No |
| PZGD-GW-35-40-01-041922 | L36997-9 | PFOS | Yes |
| PZHS-GW-6-16-01-041922 | L36997-10 | PFOS | No |
| PZHD-GW-21-26-01-041922 | L36997-11 | PFOS | No |
| PZIS-GW-7-17-01-041922 | L36997-12 | PFOS | Yes |
| PZID-GW-22-27-01-041922 | L36997-13 | PHxS | No |
| PZID-GW-22-27-01-041922 | L36997-13 | PFOS | No |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| OW20J-1-GW-310-320-01-042522 | L36997-14 | PFOS | No |
| PL41-GW-72-82-01-041922 | L36997-16 | PFOS | No |
| SP42-GW-116-126-01-041922 | L36997-17 | PFOS | No |
| W6102-GW-93-105-01-042122 | L36997-18 | PFOS | No |
| W6201-GW-109-124-01-041922 | L36997-19 | PFOS | No |
| MW1B-GW-100-120-01-041822 | L36997-20 | PFOS | No |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| PZAS-GW-6-16-01-041922 (L36997-6)<br>PZAD-GW-21-26-01-041922 (L36997-7)<br>PZHD-GW-21-26-01-041922 (L36997-11) | PFOSA | ng/L | BJ | tb |
| PZID-GW-22-27-01-041922 (L36997-13) | PFDoA<br>PFTeDA<br>N-MeFOSA<br>N-EtFOSA<br>N-MeFOSE | ng/L | UJ | s |
| | N-EtFOSE | ng/L | R | s |
| W6102-GW-93-105-01-042122 (L36997-18) | PFBA | ng/L | J+ | s |
| OW20S-1-GW-190-200-01-042522 (L36997-15)<br>MW1B-GW-100-120-01-041822 (L36997-20) | N-MeFOSA | ng/L | UJ | s |
| PZAS-GW-6-16-01-041922 (L36997-6)<br>MW1B-GW-100-120-01-041822 (L36997-20) | N-MeFOSE | ng/L | UJ | s |
| TRIP-COOLER3-050422 (L36997-1)<br>PZAS-GW-6-16-01-041922 (L36997-6)<br>PZHS-GW-6-16-01-041922 (L36997-10)<br>OW20S-1-GW-190-200-01-042522 (L36997-15) | N-EtFOSE | ng/L | UJ | s |
| All Samples | N-MeFOSA | ng/L | UJ | l |
| PZAS-GW-6-16-01-041922 (L36997-6)<br>PZID-GW-22-27-01-041922 (L36997-13) | PFHxS | ng/L | J+ | q |
| PZGD-GW-35-40-01-041922 (L36997-9)<br>PZIS-GW-7-17-01-041922 (L36997-12) | PFOS | ng/L | J+ | em |
| PZAD-GW-21-26-01-041922 (L36997-7)<br>PZGS-GW-20-30-01-041922 (L36997-8)<br>PZHS-GW-6-16-01-041922 (L36997-10)<br>PZHD-GW-21-26-01-041922 (L36997-11)<br>PZID-GW-22-27-01-041922 (L36997-13)<br>OW20J-1-GW-310-320-01-042522 (L36997-14)<br>PL41-GW-72-82-01-041922 (L36997-16 N)<br>SP42-GW-116-126-01-041922 (L36997-17)<br>W6102-GW-93-105-01-042122 (L36997-18)<br>W6201-GW-109-124-01-041922 (L36997-19)<br>MW1B-GW-100-120-01-041822 (L36997-20) | PFOS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

| | |
|---|---|
| B | Blank contamination |
| J | Estimated (+/- indicates high or low bias) |
| R | Rejected |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| em | Estimated maximum concentration |
| l | Laboratory control sample |
| q | Quantitation |
| s | Labeled compound |
| tb | Trip blank |

**Attachment A**

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG81606 (WG81251)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/11/22 23:10 | NFDHA | 167% | 70-130% | NFDHA results were nondetect, and were acceptable without qualification. |
| CCV 6/12/22 6:59 | NFDHA | 134% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Three results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-050422 | L36996-1 | D3-N-MeFOSA | 46.5 | UJ |
| PZGD-GW-35-40-02-041922 | L36996-6 | d7-NMe-FOSE | 45.3 | UJ |
| PZGD-GW-35-40-02-041922 | L36996-6 | D3-N-MeFOSA | 44.7 | UJ |
| MW5A-GW-210-220-01-042522 | L36996-11 | 13C4-PFBA | 3.85 | R |
| MW5A-GW-210-220-01-042522 | L36996-11 | 13C5-PFPeA | 44.3 | J+ |
| OW5J-1-GW-230-240-01-042122 | L36996-14 | d7-NMe-FOSE | 43.8 | UJ |
| OW5J-1-GW-230-240-01-042122 | L36996-14 | D3-N-MeFOSA | 41.4 | UJ |
| OW5J-2-GW-215-225-01-042122 | L36996-15 | 13C4-PFBA | 15.8 | R |
| OW5O-1-GW-200-210-01-042122 | L36996-17 | 13C4-PFBA | 31.2 | J+ |
| OW20J-2-GW-310-320-01-042522 | L36996-19 | 13C4-PFBA | 32.2 | J+ |
| OW20J-3-GW-280-290-01-042722 | L36996-20 | 13C4-PFBA | 8.14 | R |
| OW20J-3-GW-280-290-01-042722 | L36996-20 | d7-NMe-FOSE | 43.6 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81251-103 (Low Level) | N-MeFOSA | 64.5 | 70-130 | The associated results (all samples) were nondetect, and qualified as estimated (UJ). |
| | N-EtFOSA | 60.1 | 70-130 | |
| | N-MeFOSE | 62.6 | 70-130 | |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZGD-GW-35-40-02-041922 | L36996-6 | PFOS |
| MW17C-GW-38.5-48.5-03-042122 | L36996-10 | PFNA |
| MW5A-GW-210-220-01-042522 | L36996-11 | PFOS |
| MW5A-GW-210-220-01-042522 | L36996-11 | PFPeS |
| OW20J-3-GW-280-290-01-042722 | L36996-20 | PFOS |

The PFOS result for sample PZFS-GW-18-28-01-041922 (L36996-4) was reported from a dilution analysis due to the high sample concentration.

The laboratory confirmed that the laboratory report title page (page 2) incorrectly listed the SDG as DPWG8160**1**, and that the correct Data Package ID is DPWG8160**6**.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81606 (WG81251) Report date (mm/dd/yyyy): 6/23/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/26/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 9.3 and 8.0°C, and were above the ≤ 6 °C criteria. Results were not qualified as they were received at temperatures ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-050422<br>TRIP-COOLER2-050422 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 54.1 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | No sample detects |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | Two MS/MSDs were requested per CoC, but not reported due to a laboratory oversight:<br>PZED-GW-16-21-03-042122<br>MW17C-GW-38.5-48.5-03-042122 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Triplicates: PZED-GW-16-21-01-042122 / PZED-GW-16-21-02-042122 / PZED-GW-16-21-03-042122<br>MW17C-GW-38.5-48.5-01-042122 / MW17C-GW-38.5-48.5-02-042122 / MW17C-GW-38.5-48.5-03-042122<br>Field Duplicate: PZGD-GW-35-40-01-041922 . PZGD-GW-35-40-02-041922 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | Parent or duplicates were sometimes reported in other SDGs (WG81180, WG81182 and WG81196) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | RPDs for field triplicate/duplicate - ok<br>LCSD - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>A reduced sample volume was used for sample MW5B-GW-110-120-01-042122 (L36996-12) due to a high level of particulates.<br>A PFOS dilution was required for sample PZFS-GW-18-28-01-041922 sample (L36996-4) due to a high sample concentration. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications. Three results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/26/2022

**SGS AXYS DPWG81620** 9/25/22 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | | | | | |
|---|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | | p 1276 |
| | | | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 | | |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 | | |
| 27916 | 6263 | 2 | 1.1287 | 1.13 | | |
| 23878 | 13307 | 5 | 1.1215 | 1.11 | | |
| 24247 | 28296 | 10 | 1.1742 | 1.17 | | |
| 26274 | 63187 | 20 | 1.2099 | 1.21 | | |
| 26621 | 144547 | 50 | 1.0927 | 1.09 | | |
| 25106 | 683620 | 250 | 1.0959 | 1.09 | | |
| | | Avg: | 1.1373 | 1.13 | 1.13401 | (raw data) |
| | | %RSD: | 5.9989 | 6.15 | | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW1C-GW-40-45-01-041822 | PFOS | 281174 | 18368 | 1 | 271.65 | 272 | 0.500 | 0.004 | L36997-21 |
| EP17C-WAT-BULK-01-042122 | PFOS | 525880 | 16957 | 1 | 566.20 | 566 | 0.4860 | 0.004 | L36997-40 |
| WG81197-102 | PFOS | 11019 | 20401 | 1 | 4.79 | 4.79 | 1.00 | 0.004 | |
| WG81197-103 | PFOS | 3287 | 20317 | 1 | 1.44 | 1.44 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/10/22 10:24 | 15110 | 28350 | 4.729 | 4.73 | 5.00 | 94.6 | 94.6% |
| CCV 6/10/22 13:53 | 17901 | 35994 | 4.413 | 4.41 | 5.00 | 88.2 | 88.3% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG81197-101): | PFOS | 0.176 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG81620 (Analysis WG81197) dated 6/24/2022 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 9/26/2022 |

The samples listed below were collected at the Project 1007 Site on April 18 through 25, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| MW1C-GW-40-45-01-041822 | | 4/18/2022 | L36997-21 |
| MW2D-GW-7-17-01-041822 | | 4/18/2022 | L36997-22 |
| MW2E-GW-90-100-01-041822 | | 4/18/2022 | L36997-23 |
| MW7A-GW-200-210-01-041922 | | 4/19/2022 | L36997-24 |
| MW9A-GW-140-150-01-041822 | | 4/18/2022 | L36997-25 |
| MW9B-GW-90-100-01-041822 | | 4/18/2022 | L36997-26 |
| MW10C-GW-10-20-01-042122 | | 4/21/2022 | L36997-27 |
| MW14A-GW-316-326-01-041822 | | 4/18/2022 | L36997-28 |
| MW14B-GW-60-70-01-041822 | | 4/18/2022 | L36997-29 |
| MW14C-GW-16-36-01-041822 | | 4/18/2022 | L36997-30 |
| MW14D-GW-6-21-01-041822 | | 4/18/2022 | L36997-31 |
| MW17A-GW-230-240-01-042522 | | 4/25/2022 | L36997-32 |
| MW17B-GW-90-100-01-042522 | | 4/25/2022 | L36997-33 |
| RC3-WAT-BULK-01-041822 | | 4/18/2022 | L36997-34 |
| RC5-WAT-BULK-01-041822 | | 4/18/2022 | L36997-35 |
| RC23-WAT-BULK-01-041822 | | 4/18/2022 | L36997-36 |
| RC12-WAT-BULK-01-041822 | | 4/18/2022 | L36997-37 |
| EP17A-WAT-BULK-01-042122 | | 4/21/2022 | L36997-38 |
| EP17-WAT-BULK-01-042122 | | 4/21/2022 | L36997-39 |
| EP17C-WAT-BULK-01-042122 | | 4/21/2022 | L36997-40 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), December 2021.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✗ | Initial calibration/continuing calibration verification (CCVs) |
| ✗ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control samples/laboratory control sample duplicates (LCS/LCSDs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of one result that was rejected (R) due to a severely low labeled compound recovery. Eight PFOSA results were qualified BJ/B due to trip contamination. In addition, results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to CCVs, labeled compound recoveries, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

Samples were received at the laboratory intact. The samples were received above 6 °C at temperatures of 8.6, 9.1, and 9.2 °C. Results were not qualified as the samples were received at temperatures ≤ 10 °C.

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

## Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA (highest 2 standards), and PFUnA from the initial calibration curve due to non-linearity at the high concentrations.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point, and this calibration point was not used during calibration. In addition, the NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/9/22 23:57 | NFDHA | 163 | 70-130 | NFDHA results were nondetect and were qualified as estimated (UJ). |
| CCV 6/10/22 5:36 | NFDHA | 67.1 | 70-130 | |
| CCV 6/10/22 8:13 | NFDHA | 146 | 70-130 | |
| CCV 6/10/22 10:24 | NFDHA | 67.5 | 70-130 | |
| CCV 6/16/22 13:53 | NFDHA | 138 | 70-130 | NFDHA results not reported from this CCV. |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Two trip blanks were associated with the samples and the trip blank results were reported in DPWG81551 (WG81196). The trip blank detections are summarized in the table below. Associated sample detects within 10 times the blank concentration were qualified B/BJ.

| Trip Blank | Analyte | Concentration (ng/L) | Qualifiers |
|---|---|---|---|
| TRIP-COOLER5-050422 | PFOSA | 0.498 J | Associated sample detects were qualified B/BJ:<br><br>MW2E-GW-90-100-01-041822 (L36997-23)<br>MW10C-GW-10-20-01-042122 (L36997-27)<br>MW14B-GW-60-70-01-041822 (L36997-29)<br>MW14C-GW-16-36-01-041822 (L36997-30)<br>MW14D-GW-6-21-01-041822 (L36997-31)<br>EP17A-WAT-BULK-01-042122 (L36997-38)<br>EP17-WAT-BULK-01-042122 (L36997-39)<br>EP17C-WAT-BULK-01-042122 (L36997-40) |

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled compounds are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2E-GW-90-100-01-041822 | L36997-23 | 13C4-PFBA | 13.2 | J+ |
| MW2E-GW-90-100-01-041822 | L36997-23 | d7-NMe-FOSE | 48.5 | UJ |
| MW7A-GW-200-210-01-041922 | L36997-24 | 13C4-PFBA | 46.4 | J+ |
| MW9A-GW-140-150-01-041822 | L36997-25 | 13C4-PFBA | 17.8 | J+ |
| MW14A-GW-316-326-01-041822 | L36997-28 | 13C4-PFBA | 46.9 | J+ |
| MW17A-GW-230-240-01-042522 | L36997-32 | 13C4-PFBA | 29.2 | J+ |
| RC5-WAT-BULK-01-041822 | L36997-35 | 13C4-PFBA | 35.5 | J+ |
| RC23-WAT-BULK-01-041822 | L36997-36 | 13C4-PFBA | 31.3 | J+ |
| RC12-WAT-BULK-01-041822 | L36997-37 | 13C4-PFBA | 7.53 | R |
| EP17A-WAT-BULK-01-042122 | L36997-38 | 13C4-PFBA | 25.4 | J+ |
| EP17A-WAT-BULK-01-042122 | L36997-38 | d9-NEt-FOSE | 49.2 | UJ |
| EP17-WAT-BULK-01-042122 | L36997-39 | 13C4-PFBA | 44.5 | J+ |
| EP17C-WAT-BULK-01-042122 | L36997-40 | d9-NEt-FOSE | 49.9 | UJ |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

**LCS/LCSD Results**

LCS/LCSDs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits, except as listed in the table below.

| LCS IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | LCS RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG81197-102 WG81197-105 | NFDHA | 83.6/128 | 70-130 | **42.2** | 30 | NFDHA results were nondetect and were acceptable without qualification. |
| WG81197-103 (Low Level) | NFDHA | **138**/-- | 70-130 | **--** | 30 | |

Bold indicates an exceedance

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Eleven samples were analyzed at multiple dilutions due to high analyte concentrations.

Sample MW17B-GW-90-100-01-042522 (L36997-33) was reanalyzed for PFOS to confirm possible carry-over. The re-analysis data showed that the original PFOS analysis was affected by carry over and the re-analysis results were reported.

A smaller sample volume was used for sample prep for samples MW9B-GW-90-100-01-041822 (L36997-26) and MW17B-GW-90-100-01-042522 (L36997-33) due to the high particulate content.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and were qualified as estimated biased high (J+). PFOS qualifications are mostly attributed to ion ratio exceedances of PFOS-IV which is present at a very low concentration, but does not meet ion ratio criteria.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW1C-GW-40-45-01-041822 | L36997-21 | PFOS | No |
| MW2D-GW-7-17-01-041822 | L36997-22 | PFOS | No |
| MW2E-GW-90-100-01-041822 | L36997-23 | PFHxS | No |
| MW7A-GW-200-210-01-041922 | L36997-24 | PFHxS | No |
| MW7A-GW-200-210-01-041922 | L36997-24 | PFOS | Yes |
| MW9A-GW-140-150-01-041822 | L36997-25 | PFNS | Yes |
| MW9A-GW-140-150-01-041822 | L36997-25 | PFOS | No |
| MW9B-GW-90-100-01-041822 | L36997-26 | PFOS | No |
| MW10C-GW-10-20-01-042122 | L36997-27 | PFOS | Yes |
| MW14A-GW-316-326-01-041822 | L36997-28 | PFOS | No |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW14B-GW-60-70-01-041822 | L36997-29 | PFHxS | No |
| MW14B-GW-60-70-01-041822 | L36997-29 | EtFOSAA | No |
| MW14B-GW-60-70-01-041822 | L36997-29 | PFOS | No |
| MW14C-GW-16-36-01-041822 | L36997-30 | PFOS | No |
| MW14D-GW-6-21-01-041822 | L36997-31 | PFOS | No |
| RC3-WAT-BULK-01-041822 | L36997-34 | PFOS | No |
| RC5-WAT-BULK-01-041822 | L36997-35 | PFOS | No |
| RC23-WAT-BULK-01-041822 | L36997-36 | EtFOSAA | No |
| RC23-WAT-BULK-01-041822 | L36997-36 | PFOS | No |
| RC12-WAT-BULK-01-041822 | L36997-37 | PFOS | No |
| EP17A-WAT-BULK-01-042122 | L36997-38 | PFOS | No |
| EP17-WAT-BULK-01-042122 | L36997-39 | PFOS | No |
| EP17C-WAT-BULK-01-042122 | L36997-40 | PFOS | No |
| EP17C-WAT-BULK-01-042122 | L36997-40 | EtFOSAA | No |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All Samples | NFDHA | ng/L | UJ | c |
| MW2E-GW-90-100-01-041822 (L36997-23)<br>MW10C-GW-10-20-01-042122 (L36997-27)<br>MW14B-GW-60-70-01-041822 (L36997-29)<br>MW14C-GW-16-36-01-041822 (L36997-30)<br>MW14D-GW-6-21-01-041822 (L36997-31)<br>EP17A-WAT-BULK-01-042122 (L36997-38)<br>EP17-WAT-BULK-01-042122 (L36997-39)<br>EP17C-WAT-BULK-01-042122 (L36997-40) | PFOSA | ng/L | BJ | tb |
| MW2E-GW-90-100-01-041822 (L36997-23)<br>MW7A-GW-200-210-01-041922 (L36997-24)<br>MW9A-GW-140-150-01-041822 (L36997-25)<br>MW14A-GW-316-326-01-041822 (L36997-28)<br>MW17A-GW-230-240-01-042522 (L36997-32 N)<br>RC5-WAT-BULK-01-041822 (L36997-35)<br>RC23-WAT-BULK-01-041822 (L36997-36)<br>EP17A-WAT-BULK-01-042122 (L36997-38)<br>EP17-WAT-BULK-01-042122 (L36997-39) | PFBA | ng/L | J+ | s |
| RC12-WAT-BULK-01-041822 (L36997-37) | PFBA | ng/L | R | s |
| MW2E-GW-90-100-01-041822 (L36997-23) | N-MeFOSE | ng/L | UJ | s |
| EP17A-WAT-BULK-01-042122 (L36997-38)<br>EP17C-WAT-BULK-01-042122 (L36997-40) | N-EtFOSE | ng/L | UJ | s |
| MW7A-GW-200-210-01-041922 (L36997-24)<br>MW10C-GW-10-20-01-042122 (L36997-27) | PFOS | ng/L | J+ | em |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW9A-GW-140-150-01-041822 (L36997-25) | PFNS | ng/L | J+ | em |
| MW2E-GW-90-100-01-041822 (L36997-23)<br>MW7A-GW-200-210-01-041922 (L36997-24)<br>MW14B-GW-60-70-01-041822 (L36997-29) | PFHxS | ng/L | J+ | q |
| MW1C-GW-40-45-01-041822 (L36997-21)<br>MW2D-GW-7-17-01-041822 (L36997-22)<br>MW9A-GW-140-150-01-041822 (L36997-25)<br>MW9B-GW-90-100-01-041822 (L36997-26)<br>MW14A-GW-316-326-01-041822 (L36997-28)<br>MW14B-GW-60-70-01-041822 (L36997-29)<br>MW14C-GW-16-36-01-041822 (L36997-30)<br>MW14D-GW-6-21-01-041822 (L36997-31)<br>RC3-WAT-BULK-01-041822 (L36997-34)<br>RC5-WAT-BULK-01-041822 (L36997-35)<br>RC23-WAT-BULK-01-041822 (L36997-36)<br>RC12-WAT-BULK-01-041822 (L36997-37)<br>EP17A-WAT-BULK-01-042122 (L36997-38)<br>EP17-WAT-BULK-01-042122 (L36997-39)<br>EP17C-WAT-BULK-01-042122 (L36997-40) | PFOS | ng/L | J+ | q |
| MW14B-GW-60-70-01-041822 (L36997-29)<br>RC23-WAT-BULK-01-041822 (L36997-36)<br>EP17C-WAT-BULK-01-042122 (L36997-40) | N-EtFOSAA | ng/L | J+ | q |

(1): Data Validation Qualifiers:

| | |
|---|---|
| B | Blank contamination |
| J | Estimated (+/- indicates high or low bias) |
| R | Rejected |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| c | Calibration |
| em | Estimated maximum concentration |
| q | Quantitation |
| s | Labeled compound |
| tb | Trip blank |

**Attachment A**

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81718 (WG81247) Report date (mm/dd/yyyy): 7/11/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/27/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER5-042722 (L36954-5) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | WG81247-103 (Low Level):<br>NFDHA 150%<br>Associated results were nondetect and were acceptable without qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix<br>LCS/LCSD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/27/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG81743 (WG81249)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/11/22 14:40 | NFDHA | 142% | 70-130% | NFDHA results were nondetect and were acceptable without qualification. |
| CCV 6/11/22 18:36 | NFDHA | 144% | 70-130% | |
| CCV 6/11/22 21:25 | NFDHA | 153% | 70-130% | |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery and relative percent difference (RPD) exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG81249-102 WG81249-104 | NFDHA | **168**/113 | 70-130 | **39** | 30 | The associated results (all samples) were nondetect, and were acceptable without qualification. |
| WG81249-103 (low level) | PFDoS | **67.5/--** | 70-130 | **--** | -- | The associated results (all samples) were nondetect and qualified as estimated (UJ). |
| | N-MeFOSA | **62.1/--** | 70-130 | **--** | -- | |
| | N-EtFOSA | **68.2/--** | 70-130 | **--** | -- | |
| | NFDHA | **140/--** | 70-130 | **--** | -- | The associated results (all samples) were nondetect, and were acceptable without qualification. |

**Bold** indicates an exceedance

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81743 (WG81249) Report date (mm/dd/yyyy): 7/12/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/3/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature 6.3°C, and was above the ≤ 6 °C criteria. Results were not qualified as the samples were received at a temperature of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. No qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | TRIP-COOLER1-052522 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for water samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix<br>LCS/LCSD RPDs:<br>NFDHA 39%<br>No qualifications (results were nondetect). |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/4/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG81745 (WG81367)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/23/22 4:27 | NFDHA | 168% | 70-130% | NFDHA results were nondetect, and were acceptable without qualification. |
| CCV 6/24/22 18:22 | PFHpS | 0.2% | 70-130% | PFHpS was not reported from this CCV. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW19-SED-0-12-02-051922 | L37128-21 | D3-N-MeFOSA | 49.1 | J+ |
| IW19-SED-0-12-02-051922 | L37128-21 | D5-N-EtFOSA | 46.7 | UJ |
| IW20-SED-0-12-01-051922 | L37128-23 | D5-N-EtFOSA | 48.5 | UJ |
| IW25-SED-0-12-01-051922 | L37128-25 | D5-N-EtFOSA | 46.3 | J+ |
| IW26-SED-0-12-01-051922 | L37128-26 | D5-N-EtFOSA | 44.4 | UJ |
| IW26-SED-0-12-02-051922 | L37128-27 | D3-N-MeFOSA | 29.1 | J+ |
| IW26-SED-0-12-02-051922 | L37128-27 | D5-N-EtFOSA | 23.2 | UJ |
| IW26-SED-0-12-02-051922 | L37128-27 | d7-NMe-FOSE | 42.0 | UJ |
| IW27-SED-0-12-01-051922 | L37128-28 | D3-N-MeFOSA | 39.6 | J+ |
| IW27-SED-0-12-01-051922 | L37128-28 | D5-N-EtFOSA | 36.6 | UJ |
| IW31-SED-0-12-01-051922 | L37128-29 | D5-N-EtFOSA | 46.0 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Laboratory control sample recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81367-102 | NFDHA | 139 | 70-130 | The associated results (all samples) were nondetect, and did not require qualification due to LCS recoveries. |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81367-103 (Low Level) | N-EtFOSA | 60.2 | 70-130 | Associated detects previously qualified J+ were updated to a J qualifier, as both high and low bias are present. The detect for sample IW24-SED-0-12-01-051922 (L37128-24) was qualified as estimated biased low (J-). Nondetects were qualified UJ. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Qualification was not required.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **IW19-SED-0-12-03-051922:** | | | |
| N-MeFOSA | **153**/89.6 | 70-130 | The detects for samples IW19-SED-0-12-02-051922 and IW19-SED-0-12-03-051922 were qualified as estimated biased high (J+). |
| PFOS | Not Reported | -- | The sample concentration was greater than four times the spike concentration. No qualifiers. |

Results qualified as estimated (J) due to field imprecision are listed below, as the results were greater than ± 2 times the reporting limit (RL).

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Precision Limit |
|---|---|---|---|
| **IW26-SED-0-12-01-051922 / IW26-SED-0-12-02-051922:** | | | |
| EtFOSAA | 0.163 | 0.335 | > ± 2x RL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW24-SED-0-12-01-051922 | L37128-24 | N-EtFOSA |
| IW25-SED-0-12-01-051922 | L37128-25 | N-EtFOSA |
| IW25-SED-0-12-01-051922 | L37128-25 | N-MeFOSA |
| IW26-SED-0-12-02-051922 | L37128-27 | N-MeFOSAA |

Nine PFOS results were reported from a dilution analysis due to the high sample concentrations.

The lab report file name was DPWG81745; however, the laboratory report title page (page 2) incorrectly listed the SDG as DPWG8174, and the correlation table incorrectly listed the SDG as DPWG81744.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81745 (WG81367) Report date (mm/dd/yyyy): 7/11/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/31/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature 6.3°C, and above the ≤ 6 °C criteria. Results were not qualified as the samples were received at a temperature of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-052522 was received with the samples (reported in WG81249) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for sediment samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW19-SED-0-12-03-051922 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Triplicates: IW19-SED-0-12-01-051922 / IW19-SED-0-12-02-051922 / IW19-SED-0-12-03-051922 (Parent reported in WG81366)<br>Field Duplicate: IW26-SED-0-12-01-051922 / IW26-SED-0-12-02-051922 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | RPDs for field triplicate/duplicate - 50% RPD (or ± 2x RL for detects within 5x the RL) used for sediment matrix.<br>See data qualifications summary for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting.<br>PFOS results were reported from dilution analyses for nine samples due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/31/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG81751 (WG81366)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/22/22 23:01 | NFDHA | 144% | 70-130% | NFDHA results were nondetect and were acceptable without qualification. |
| CCV 6/24/22 13:34 | NFDHA | 131% | 70-130% | |
| CCV 6/24/22 13:34 | N-EtFOSA | 66.2% | 70-130% | N-EtFOSA results were not reported from these CCVs. No qualifiers. |
| CCV 6/24/22 16:11 | N-EtFOSA | 67.5% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| IW06-SED-0-12-01-051822 | L37128-1 | D5-N-EtFOSA | 49.0 | UJ |
| IW10-SED-0-12-01-051822 | L37128-2 | D5-N-EtFOSA | 48.1 | UJ |
| IW10-SED-0-12-02-051822 | L37128-3 | D5-N-EtFOSA | 48.1 | UJ |
| IW16-SED-0-12-01-051822 | L37128-5 | D5-N-EtFOSA | 43.6 | UJ |
| IW28-SED-0-12-01-051822 | L37128-6 | D5-N-EtFOSA | 47.5 | UJ |
| IW35-SED-0-12-01-051822 | L37128-7 | D5-N-EtFOSA | 41.5 | UJ |
| IW37-SED-0-12-01-051822 | L37128-8 | D5-N-EtFOSA | 46.1 | UJ |
| IW39-SED-0-12-01-051822 | L37128-9 | D5-N-EtFOSA | 39.8 | J+ |
| IW12-SED-0-12-01-051922 | L37128-18 | D5-N-EtFOSA | 43.4 | UJ |
| IW15-SED-0-12-01-051922 | L37128-19 | D5-N-EtFOSA | 47.3 | UJ |
| IW19-SED-0-12-01-051922 | L37128-20 | D5-N-EtFOSA | 47.7 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81366-103 (low level) | N-MeFOSA | 60.7 | 70-130 | Associated detects were qualified as estimated biased low (J-) and nondetects were qualified UJ. |
| | NFDHA | 150 | 70-130 | The associated results (all samples) were nondetect, and were acceptable without qualification. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below. Results did not require qualification.

| Compound | MS/MSD % Recovery | Recovery Limits | Results Qualified |
|---|---|---|---|
| **IW10-SED-0-12-03-051822:** | | | |
| PFOS | 1170/889 | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| IW10-SED-0-12-01-051822 | L37128-2 | PFPeS |
| IW10-SED-0-12-01-051822 | L37128-2 | N-MeFOSAA |
| IW16-SED-0-12-01-051822 | L37128-5 | N-MeFOSA |
| IW45-SED-0-12-01-051822 | L37128-11 | PFDoA |
| IW45-SED-0-12-01-051822 | L37128-11 | N-MeFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG81751 (WG81366) Report date (mm/dd/yyyy): 7/6/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/10/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature 6.3°C, and were above the ≤ 6 °C criteria. Results were not qualified as the samples were received at a temperature of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. No qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-052522 (reported in DPWG81743) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | IW10-SED-0-12-03-051822 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☒ | ☐ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☒ | ☐ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☒ | ☐ | ☐ | Re-ran to bring the PFOS sample concentration within calibration range. |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field triplicate: IW10-SED-0-12-01-051822 / IW10-SED-0-12-02-051822 / IW10-SED-0-12-03-051822 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | 50% RPD limit used for sediment matrix<br>RPDs - ok. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | Laboratory J flags indicating a concentration less than limit of quantification are to be retained during reporting. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | PFOS result for eleven samples were reported from dilution analyses due to high sample concentrations. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/10/2022

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82286 (WG82092) Report date (mm/dd/yyyy): 10/24/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/22/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 11.4 °C, and above the ≤ 6 °C criteria. The results were nondetects and qualified as estimated (UJ), as the samples were received at a temperature greater than 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | The associated trip blank was not reported in this SDG:<br>TRIP-COOLER2-062222 (L37348-2)<br>Reported in SDG DPWG82367 (WG82147) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW22B-SED-68-70-01-060622 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 50% RPD limit used for sediment matrix |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☐ | ☒ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/22/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82286 (WG82092)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature of 11.4 °C, and above the ≤ 6 °C criteria. The results were nondetect and qualified as estimated (UJ), as the samples were received at a temperature greater than 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ). Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW22B-SED-68-70-01-060622 | L37353-1 (A) | D3-N-MeFOSA | 46.2 | UJ |
| MW22B-SED-68-70-01-060622 | L37353-1 (A) | D3-MeFOSAA | 47.4 | UJ |
| MW21A-SED-COMP-76-80-01-061422 | L37353-2 | D3-N-MeFOSA | 46.7 | UJ |
| MW21A-SED-COMP-76-80-01-061422 | L37353-2 | D3-MeFOSAA | 47.3 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82092-102 | NFDHA | 59.8 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| WG82092-103 (Low Level) | N-MeFOSA | 55.0 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| | N-EtFOSA | 65.3 | 70-130 | |
| | 11Cl-PF3OUdS | 69.3 | 70-130 | |
| | 7:3 FTCA | 65.5 | 70-130 | |
| | NFDHA | 59.6 | 70-130 | |

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82286 (WG82092)**

**Chain of custody preservation, and holding time (Section 1C):** All of the samples were received at a temperature of 11.4 °C. Results were nondetect and qualified as estimated (UJ) as they were received at a temperature ≥ 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ), as summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW22B-SED-68-70-01-060622 | L37353-1 (A) | D3-N-MeFOSA | 46.2 | UJ |
| MW22B-SED-68-70-01-060622 | L37353-1 (A) | D5-N-EtFOSA | 47.4 | UJ |
| MW21A-SED-COMP-76-80-01-061422 | L37353-2 | D3-N-MeFOSA | 46.7 | UJ |
| MW21A-SED-COMP-76-80-01-061422 | L37353-2 | D5-N-EtFOSA | 47.3 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82092-102 | NFDHA | 59.8 | 70-130 | The associated results (all samples) were nondetect, and were qualified UJ. |
| WG82092-103 (low level) | N-MeFOSA | 55.0 | 70-130 | The associated results (all samples) were nondetect, and were qualified UJ. |
| | N-EtFOSA | 65.3 | 70-130 | |
| | 11Cl-PF3OUdS | 69.3 | 70-130 | |
| | 7:3 FTCA | 65.5 | 70-130 | |
| | NFDHA | 59.6 | 70-130 | |

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82286 (WG82092) Report date (mm/dd/yyyy): 10/24/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/25/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 11.4°C, and above the ≤ 6 °C criteria. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER2-062222 (reported in DPWG82367) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW22B-SED-68-70-01-060622 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 50% RPD limit used for sediment matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☐ | ☒ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/25/2022

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82367 (WG82147) Report date (mm/dd/yyyy): 8/22/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/24/2022

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 10.2 and 11.4°C, and were above the ≤ 6 °C criteria. Detects for these samples were qualified as estimated biased low (J-) and nondetects were qualified UJ as they were received at temperatures ≥ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-062222<br>TRIP-COOLER2-062222<br>EQ-DRILLWAT-01-060722<br>EQ-DRILLPUMP-01-062122 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | EQ-DRILLWAT-01-060722:<br>PFOSA 1.01 J ng/L<br>EQ-DRILLPUMP-01-062122:<br>PFOS 1.87 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | Only field QC blanks were reported in this SDG. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/24/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82496 (WG81945)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/22/22 20:50 | NFDHA | 183% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 8/23/22 00:47 | N-EtFOSE | 136% | 70-130% | |
| | NFDHA | 157% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ), as summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW3B-GW-110-130-01-071522 | L37502-78 | D5-N-EtFOSA | 37.9 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG81945-102 WG81945-104 | NFDHA | 178/188 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG81945-103 (low level) | N-EtFOSA | 61.1/-- | 70-130 | The associated results (all samples) were nondetect, and were qualified UJ. |
| | NFDHA | 220/-- | 70-130 | The associated results were nondetect and were acceptable without qualification. |

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# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82496 (WG81945) Report date (mm/dd/yyyy): 9/7/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/24/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | TRIP-COOLER7-072022 (reported in WG82115) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | A reduced sample volume was used for MW3B-GW-110-130-01-071522 (L37502-78) due to high suspended solids content. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/24/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82616 (DPWG82616)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/31/22 17:20 | NFDHA | 177% | 70-130% | The associated result was nondetect and was acceptable without qualification. |
| CCV 8/31/22 21:03 | NFDHA | 215% | 70-130% | |
| CCV 8/31/22 23:54 | NFDHA | 229% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were positive results and were qualified as estimated biased high (J+), as the labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26A-WAT-12-01-071822 | L37502-12 R | 13C4-PFBA | 40.2 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82128-102 WG82128-107 | NFDHA | 196/200 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82128-103 (low level) | N-MeFOSA | 60.4/-- | 70-130 | The associated results were nondetect, and were qualified UJ. |
| | N-EtFOSA | 66.8/-- | 70-130 | |
| | NFDHA | 301/-- | 70-130 | The associated results were nondetect and were acceptable without qualification. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82616 (WG82128) Report date (mm/dd/yyyy): 9/14/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/25/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-072022 (reported in WG82115) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | PFOSA 0.609 BJ ng/L<br>This trip blank detection was determined to be laboratory contamination. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/25/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82631 (WG82117)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at temperatures of 0.7, 3.7 and 10.7 °C. Detects for samples received at 10.7 °C were qualified as estimated biased low (J-) and nondetects were qualified UJ, as they were received at a temperature ≥ 10 °C.

| Sample ID | Lab ID |
|---|---|
| PL41-GW-72-82-01-071122 | L37502-17 |
| PL41-GW-72-82-02-071122 | L37502-18 |
| PL41-GW-72-82-03-071122 | L37502-19 |
| W6102-GW-93-105-01-071322 | L37502-20 |
| W6201-GW-109-124-01-071322 | L37502-21 |
| OD3-WAT-BULK-01-071822 | L37502-22 |
| OD4-WAT-BULK-01-071822 | L37502-23 |
| OD5-WAT-BULK-01-071822 | L37502-24 |
| OD12-WAT-BULK-01-071822 | L37502-25 |
| RC5-WAT-BULK-01-071822 | L37502-26 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/26/22 3:03 | NFDHA | 242% | 70-130% | The associated results were nondetect and was acceptable without qualification. |
| CCV 8/26/22 6:32 | NFDHA | 198% | 70-130% | |
| CCV 8/26/22 8:57 | NFDHA | 141% | 70-130% | |
| CCV 8/26/22 10:42 | NFDHA | 248% | 70-130% | |
| CCV 8/30/22 5:27 | NFDHA | 224% | 70-130% | |
| CCV 8/30/22 7:51 | NFDHA | 229% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results were rejected (R) due to severely low recoveries. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26A-WAT-12-24-01-070522 | L37502-16 | d9-NEt-FOSE | 35.9 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26A-WAT-12-24-01-070522 | L37502-16 | 13C2-PFTeDA | 42.3 | UJ<br>PFTrDA: UJ |
| EP26A-WAT-12-24-01-070522 | L37502-16 | D5-N-EtFOSA | 35.7 | UJ |
| PL41-GW-72-82-01-071122 | L37502-17 | 13C4-PFBA | 38.6 | J+ |
| PL41-GW-72-82-02-071122 | L37502-18 | 13C4-PFBA | 42.4 | J+ |
| W6201-GW-109-124-01-071322 | L37502-21 | d9-NEt-FOSE | 44.3 | UJ |
| W6201-GW-109-124-01-071322 | L37502-21 | D3-MeFOSAA | 47.3 | UJ |
| W6201-GW-109-124-01-071322 | L37502-21 | D5-EtFOSAA | 48.5 | UJ |
| W6201-GW-109-124-01-071322 | L37502-21 | 13C5-PFHxA | 49.8 | J+ |
| W6201-GW-109-124-01-071322 | L37502-21 | D3-N-MeFOSA | 46.6 | UJ |
| W6201-GW-109-124-01-071322 | L37502-21 | D5-N-EtFOSA | 38.0 | UJ |
| EP2A-WADE-WAT-BULK-01-071822 | L37502-27 | 13C2-4:2 FTS | 157 | No Qualifier |
| EP18E-WAT-BULK-01-071822 | L37502-28 | 13C2-PFTeDA | 46.8 | C |
| EP18E-WADE-WAT-BULK-01-071822 | L37502-29 | d9-NEt-FOSE | 11.7 | UJ |
| EP18E-WADE-WAT-BULK-01-071822 | L37502-29 | d7-NMe-FOSE | 22.3 | UJ |
| EP18E-WADE-WAT-BULK-01-071822 | L37502-29 | 13C2-PFDoA | 47.4 | UJ |
| EP18E-WADE-WAT-BULK-01-071822 | L37502-29 | 13C2-PFTeDA | 8.67 | R<br>PFTrDA: R |
| EP18E-WADE-WAT-BULK-01-071822 | L37502-29 | D5-N-EtFOSA | 39.8 | UJ |
| EP17-WAT-BULK-01-071822 | L37502-30 | d9-NEt-FOSE | 31.7 | UJ |
| EP17-WAT-BULK-01-071822 | L37502-30 | d7-NMe-FOSE | 35.6 | UJ |
| EP17-WAT-BULK-01-071822 | L37502-30 | 13C4-PFBA | 20.1 | J+ |
| EP17-WAT-BULK-01-071822 | L37502-30 | 13C2-PFTeDA | 31.0 | UJ<br>PFTrDA: UJ |
| EP19-WAT-BULK-01-071822 | L37502-31 | 13C4-PFBA | 39.7 | J+ |
| EP19-WAT-BULK-01-071822 | L37502-31 | 13C2-PFTeDA | 49.7 | UJ<br>PFTrDA: UJ |
| EP24-WAT-BULK-01-071822 | L37502-32 | d9-NEt-FOSE | 45.6 | UJ |
| EP24-WAT-BULK-01-071822 | L37502-32 | 13C4-PFBA | 30.2 | J+ |
| EP24-WAT-BULK-01-071822 | L37502-32 | 13C2-PFTeDA | 45.7 | UJ<br>PFTrDA: UJ |
| EP26D-WAT-BULK-01-071822 | L37502-33 | d9-NEt-FOSE | 47.8 | UJ |
| EP26D-WAT-BULK-01-071822 | L37502-33 | 13C4-PFBA | 48.4 | J+ |
| EP26D-WAT-BULK-01-071822 | L37502-33 | 13C2-PFTeDA | 41.3 | UJ<br>PFTrDA: J+ |
| EP26D-WADE-WAT-BULK-01-071822 | L37502-34 | d9-NEt-FOSE | 2.67 | R |
| EP26D-WADE-WAT-BULK-01-071822 | L37502-34 | d7-NMe-FOSE | 9.69 | R |
| EP26D-WADE-WAT-BULK-01-071822 | L37502-34 | 13C4-PFBA | 47.4 | J+ |
| EP26D-WADE-WAT-BULK-01- | L37502-34 | 13C2-PFTeDA | 22.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| 071822 | | | | PFTrDA: UJ |
| EP27A-WAT-BULK-01-071822 | L37502-35 | d9-NEt-FOSE | 48.2 | UJ |
| EP27A-WAT-BULK-01-071822 | L37502-35 | 13C2-PFTeDA | 26.8 | UJ<br>PFTrDA: UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82117-102 | N-MeFOSA | 63.5 | 70-130 | The associated results were nondetect, and were qualified UJ. |
| | N-EtFOSA | 56.4 | 70-130 | |
| | NFDHA | 249 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82117-103 (low level) | N-MeFOSA | 45.5 | 70-130 | The associated results were nondetect, and were qualified UJ. |
| | N-EtFOSA | 52.3 | 70-130 | |
| | NFDHA | 166 | 70-130 | The associated results were nondetect and were acceptable without qualification. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **PL41-GW-72-82-03-071122:** | | | | | |
| PFHpA | 78.5/**68.5** | 70-130 | 3.63 | 30 | The results for samples PL41-GW-72-82-01-071122, PL41-GW-72-82-02-071122, and PL41-GW-72-82-03-071122 were qualified as estimated biased low (J-). |
| PFMPA | **60.0**/85.5 | 70-130 | 16.8 | 30 | |
| N-MeFOSA | **66.7/65.6** | 70-130 | 2.88 | 30 | The results for samples PL41-GW-72-82-01-071122, PL41-GW-72-82-02-071122, and PL41-GW-72-82-03-071122 were nondetect, and qualified UJ. |
| N-EtFOSA | **65.1/89.4** | 70-130 | **30.2** | 30 | |
| 9Cl-PF3ONS | 73.9/**68.4** | 70-130 | 8.95 | 30 | |
| 11Cl-PF3OUdS | **68.5/59.5** | 70-130 | 12.5 | 30 | |
| NFDHA | **212/263** | 70-130 | 20.2 | 30 | The results for samples PL41-GW-72-82-01-071122, PL41-GW-72-82-02-071122, and PL41-GW-72-82-03-071122 were nondetect, and was acceptable without qualification. |
| PFBA | Not Reported | 70-130 | 0.28 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFPeA | **15.6/52.5** | 70-130 | 7.54 | 30 | |
| PFHxA | Not Reported | 70-130 | 5.54 | 30 | |
| PFOA | Not Reported | 70-130 | 4.48 | 30 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OD12-WAT-BULK-01-071822 | L37502-25 | PFHpS |
| EP2A-WADE-WAT-BULK-01-071822 | L37502-27 | N-EtFOSAA |
| EP26D-WAT-BULK-01-071822 | L37502-33 | PFTrDA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82631 (WG82117) Report date (mm/dd/yyyy): 9/14/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/15/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at temperatures of 0.7, 3.7 and 10.7 °C, and nine samples were above the ≤ 6 °C criteria. Results for samples received over 10 °C were qualfiied as discussed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-072022<br>TRIP-COOLER2-072022<br>TRIP-COOLER3-072022<br>(reported in DPWG82722/WG82115) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The detect for TRIP-COOLER1-072022 was below the reporting limit:<br>PFOSA 0.609 BJ ng/L<br>This trip blank detection was determined to be laboratory contamination. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PL41-GW-72-82-03-071122 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD - See data qualifications summary (attachment) for exceedances.<br>Field Triplicate:<br>PL41-GW-72-82-01-071122<br>PL41-GW-72-82-02-071122<br>PL41-GW-72-82-03-071122 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for three samples (-21, -22, and -23) due to high solids, and a reduced sample volume was used for sample -26 due to high analyte concentrations.<br>PFHxA for sample -22 was reported from a re-analysis run due to instrument carry over.<br>PFOS results for samples -28, -31 and -33 were reported from a dilution analysis due to high sample concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications. Four results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/15/2022

**SGS AXYS DPWG81620** 1/17/23 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | p 242 |
| | | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 | |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 | |
| 27916 | 6263 | 2 | 1.1287 | 1.13 | |
| 23878 | 13307 | 5 | 1.1215 | 1.11 | |
| 24247 | 28296 | 10 | 1.1742 | 1.17 | |
| 26274 | 63187 | 20 | 1.2099 | 1.21 | |
| 26621 | 144547 | 50 | 1.0927 | 1.09 | |
| 25106 | 683620 | 250 | 1.0959 | 1.09 | |
| | | Avg: | 1.1373 | 1.13 | 1.13401 (raw data) |
| | | %RSD: | 5.9989 | 6.15 | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW21A-GW-286-290-01-061622 | PFOS | 81402 | 16258 | 1 | 638.30 | 638 | 0.0696 | 0.004 | L37347-12 R2 |
| WG82476-102 | PFOS | 12970 | 23716 | 1 | 4.85 | 4.85 | 1.00 | 0.004 | |
| WG82476-103 | PFOS | 4860 | 25758 | 1 | 1.67 | 1.67 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 9/11/22 11:14 | 15286 | 27411 | 4.948 | 4.95 | 5.00 | 99.0 | 99.0% |
| CCV 9/11/22 17:21 | 15349 | 28062 | 4.853 | 4.84 | 5.00 | 96.8 | 96.9% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG82476-101): | PFOS | 0.020 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG82708 (Analysis WG82476) dated 9/21/2022 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 12/27/2022 |

The sample listed below was collected at the Project 1007 Site on June 16, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Water Samples:** | | | |
| MW21A-GW-286-290-01-061622 | | 6/16/2022 | L37347-12 R2 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2022.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification (CCVs) |
| ✓ | Laboratory and trip blanks |
| ✓ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control samples (LCSs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample receipt temperature, LCS recoveries, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

The sample was received at the laboratory intact; however, was above the 6 °C temperature requirement at 11.4 °C. Sample detects were qualified as estimated biased low (J-) and nondetect results were qualified UJ as the sample was received at a temperature > 10 °C.

Additional samples (not reported in this SDG) were listed on the COCs.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

### Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA (highest 2 standards), and PFUnA from the initial calibration curve due to non-linearity at the high concentrations.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point, and this calibration point was not used during calibration. In addition, the NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/11/22 11:14 | NFDHA | 145 | 70-130 | The associated results were nondetect and was acceptable without qualification. |
| CCV 9/11/22 17:21 | NFDHA | 180 | 70-130 | |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. One trip blank (TRIP-COOLER2-062222, L37348-2, reported in DPWG82367) was associated with the sample data. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%.

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits, except as listed in the table below.

| LCS IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82476-102 | NFDHA | 172 | 70-130 | NFDHA results were nondetect and were acceptable without qualification. |
| WG82476-103 (Low Level) | N-EtFOSA | 67.5 | 70-130 | N-EtFOSA results were nondetect and qualified as estimated (UJ). |
| | NFDHA | 160 | 70-130 | NFDHA results were nondetect and were acceptable without qualification. |

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for one PFOS result and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

A smaller sample volume was used for sample prep for sample MW21A-GW-286-290-01-061622 (L37347-12 R2) due to due to severe clogging of the SPE cartridge.

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and are considered biased high. The PFOS qualification is attributable to an ion ratio exceedance of PFOS-IV which is present at a low concentration, but does not meet ion ratio criteria. This result was qualified as estimated (J), as both high bias and low bias are present.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW21A-GW-286-290-01-061622 | L37347-12 R2 | PFOS | No |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW21A-GW-286-290-01-061622 (L37347-12 R2) | All PFAS | ng/L | Detects: J-<br>Nondetects: UJ | sr |
| MW21A-GW-286-290-01-061622 (L37347-12 R2) | N-EtFOSA | ng/L | UJ | l |
| MW21A-GW-286-290-01-061622 (L37347-12 R2) | PFOS | ng/L | J | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

l Laboratory control sample
q Quantitation

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82721 (WG82118)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/24/22 3:54 | NFDHA | 280% | 70-130% | The associated results were nondetect and was acceptable without qualification. |
| CCV 8/24/22 7:50 | NFDHA | 264% | 70-130% | |
| CCV 8/24/22 10:28 | NFDHA | 207% | 70-130% | |
| CCV 9/2/22 15:03 | NFDHA | 186% | 70-130% | |
| CCV 9/2/22 17:00 | NFDHA | 212% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW20J-1-GW-310-320-01-071422 | L37502-39 | 13C4-PFBA | 43.0 | J+ |
| OW20J-1-GW-310-320-01-071422 | L37502-39 | d9-NEt-FOSE | 47.0 | UJ |
| OW5J-1-GW-230-240-01-071522 | L37502-45 | 13C4-PFBA | 17.0 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82118-102 | NFDHA | 209 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82118-104 (LCSD) | NFDHA | 239 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82118-103 (Low Level) | N-MeFOSA | 62.2 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). |
| | NFDHA | 169 | 70-130 | The associated results were nondetect and were acceptable without qualification. |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as

maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW5A-GW-210-220-01-071522 | L37502-41 | PFOS |
| MW5B-GW-110-120-01-071522 | L37502-42 | PFBS |
| MW18A-GW-352-362-01-071822 | L37502-43 | PFOS |
| OW5J-1-GW-230-240-01-071522 | L37502-45 | PFOS |

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82721 (WG82118) Report date (mm/dd/yyyy): 9/23/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/16/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at temperatures of 0.7, 4.7, and 9.7 °C, and some of the samples were above the ≤ 6 °C criteria. Results were not qualified as the samples were received at temperatures of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER3-072022<br>TRIP-COOLER4-072022<br>TRIP-COOLER5-072022<br>TRIP-COOLER6-072022<br>(reported in DPWG82722/WG82115) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | No method blank detect. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifiers for high recoveries (associated results were nondetect). |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD - no exceedances<br>Field Duplicate: |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | WL6-WAT-BULK-01-071822<br>WL6-WAT-BULK-02-071822 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for nine samples due to high solids content in the samples.<br>The PFOS result for sample MW18B-GW-225-235-01-071422 (SGS AXYS ID: L37502-44) was reported from a dilution analysis due to matrix interferences. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/16/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82722 (WG82115)**

**Chain of custody preservation, and holding time (Section 1C):** Some of the samples were received at a temperature of 10.7 °C, and were qualified as estimated (UJ/J-) as the temperature was greater than 10 °C.

| Sample ID | Lab ID |
|---|---|
| TRIP-COOLER2-072022 | L37502-2 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/25/22 17:26 | NFDHA | 210% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 8/25/22 21:09 | NFDHA | 207% | 70-130% | |
| CCV 8/25/22 22:40 | NFDHA | 279% | 70-130% | |

**Blank detections (Section 3Aii):** Method blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| WG82115-101 | PFOSA | 0.521 J | Associated detects were within 10 times the blank concentration, and were qualified BJ:<br><br>EP26A-WAT-0-3-01-071822 (L37502-11)<br>EP26A-WAT-24-01-071822 (L37502-13)<br>EP26A-WAT-BULK-01-062422 (L37502-14)<br>EP26A-WAT-0-3-01-070522 (L37502-15)<br>TRIP-COOLER1-072022 (L37502-1) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EQ-AUGER-01-071822 | L37502-9 | 13C4-PFBA | 180 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C5-PFPeA | 215 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C5-PFHxA | 180 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C4-PFHpA | 178 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C8-PFOA | 184 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EQ-AUGER-01-071822 | L37502-9 | 13C9-PFNA | 180 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C6-PFDA | 187 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C7-PFUnA | 182 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C2-PFDoA | 177 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C2-PFTeDA | 168 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C3-PFBS | 180 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C3-PFHxS | 185 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C8-PFOS | 193 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C2-4:2 FTS | 210 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C2-6:2 FTS | 182 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C2-8:2 FTS | 177 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C8-PFOSA | 192 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | d7-NMe-FOSE | 186 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | d9-NEt-FOSE | 164 | No Qualifier |
| EQ-AUGER-01-071822 | L37502-9 | 13C3-HFPO-DA | 215 | No Qualifier |
| EP26A-WAT-24-01-071822 | L37502-13 | 13C2-4:2 FTS | 154 | No Qualifier |
| EP26A-WAT-0-3-01-070522 | L37502-15 | 13C4-PFBA | 45.8 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82115-102 | NFDHA | 191 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82115-104 (LCSD) | NFDHA | 202 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82115-103 (Low Level) | N-MeFOSA | 53.1 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| | N-EtFOSA | 47.4 | 70-130 | |
| | NFDHA | 196 | 70-130 | The associated results were nondetect and were acceptable without qualification. |

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82722 (WG82115) Report date (mm/dd/yyyy): 9/23/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/19/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at temperatures of 0.7, 1.2, 2.5, 3.7, 4.7, 9.7, and 10.7 °C, and some of the samples were above the ≤ 6 °C criteria. Results for samples received above 10 °C were qualified as estimated (UJ/J-). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-072022<br>TRIP-COOLER2-072022<br>TRIP-COOLER3-072022<br>TRIP-COOLER4-072022<br>TRIP-COOLER5-072022<br>TRIP-COOLER6-072022<br>EQ-DIPPER-01-071822<br>EQ-AUGER-01-071822<br>EQ-TUBE-01-071822<br>Many of these blanks are associated with samples reported in other SDGs. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL:<br>TRIP-COOLER1-072022:<br>PFOSA 0.609 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | This trip blank detect was previously determined to be laboratory contamination. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>PFOSA 0.521 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifiers for high recoveries as associated results were nondetect. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/19/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82723 (WG82568)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15-WAT-BULK-01-071822 | L37502-52 | 13C4-PFBA | 30.7 | J+ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82723 (WG82568) Report date (mm/dd/yyyy): 10/5/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/19/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | TRIP-COOLER5-072022<br>This trip blanks was reported in SDG DPWG82722. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | No detects. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| | | Question | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/19/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82834 (WG82119)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 8/29/22 22:41 | NFDHA | 206% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 8/30/22 02:49 | NFDHA | 249% | 70-130% | |
| CCV 8/30/22 05:26 | NFDHA | 225% | 70-130% | |
| CCV 9/11/22 07:44 | NFDHA | 139% | 70-130% | |
| CCV 9/11/22 09:29 | NFDHA | 152% | 70-130% | |

**Blank detections (Section 3Bii):** Method blank detections and the results qualified are listed in the table below.

| Method Blank ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| WG82119-101 | PFOA | 0.522 J | Associated detects that were within 10 times the blank concentration, taking into consideration sample volumes and dilutions, were qualified B:<br><br>PZGS-GW-20-30-01-071322 (L37502-73)<br>PZGS-GW-20-30-02-071322 (L37502-74) |
| | PFOS | 12.5 | Associated detects that were within 10 times the blank concentration, taking into consideration sample volumes (dilutions), were qualified B:<br><br>MW2E-GW-90-100-01-071422 (L37502-66)<br>PZBD-GW-16-21-01-071222 (L37502-67)<br>PZES-GW-6-11-01-071222 (L37502-68)<br>PZED-GW-16-21-02-071222 (L37502-70)<br>PZFD-GW-33-38-01-071222 (L37502-72) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW14A-GW-316-326-01-071422 | L37502-59 | 13C4-PFBA | 32.1 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS/LCSD IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82119-102 | NFDHA | 175 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| WG82119-103 (Low Level) | N-EtFOSA | 62.0 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| | NFDHA | 210 | 70-130 | The associated results were nondetect and were acceptable without qualification. |

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82834 (WG82119) Report date (mm/dd/yyyy): 10/3/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 12/7/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at temperatures of 2.5 and 9.7 °C, and some of the samples were above the ≤ 6 °C criteria. Results were not qualified as the samples were received at temperatures of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER6-072022<br>TRIP-COOLER7-072022<br>(reported in SDG DPWG82722 (WG82115)) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | No detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | PFOA 0.522 J ng/L<br>PFOS 12.5 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifiers for high recoveries as associated results were nondetect. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | MS/MSD was requested for MW2D-GW-7-17-03-071222; however MS/MSD results were not reported. The laboratory indicated that it was logged in as an MS/MSD, but was missed at the analyst level. |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field triplicate;MW2D-GW-7-17-01-071222 / MW2D-GW-7-17-02-071222 / MW2D-GW-7-17-03-071222<br>Field duplicates: PZED-GW-16-21-01-071222 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | / PZED-GW-16-21-02-071222<br>PZGS-GW-20-30-01-071322 / PZGS-GW-20-30-02-071322 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for groundwater matrix<br>RPDs - ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | PFOA and PFOS were reported from a dilution analysis for five samples due to concentrations that exceeded the instrument calibration range during undiluted analysis.<br>Reduced sample volumes were used for seven samples due to high solids,resulting in elevated reporting limits. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 12/7/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82860 (WG82620)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/20/22 16:59 | NFDHA | 63.7% | 70-130% | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP21-WAT-BULK-01-071822 | L37502-55 | d9-NEt-FOSE | 43.1 | UJ |
| EP21-WAT-BULK-02-071822 | L37502-56 | D5-N-EtFOSA | 49.3 | UJ |
| MW10B-GW-130-140-01-071422 | L37502-62 | 13C4-PFBA | 16.6 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG82620-102 | NFDHA | 67.3 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| WG82620-103 (LowLevel) | N-MeFOSA | 50.1 | 70-130 | The associated results were nondetect and qualified as estimated (UJ). All samples were associated with this LCS. |
| | N-EtFOSA | 41.9 | 70-130 | |
| | N-EtFOSE | 67.7 | 70-130 | |

**MS/MSD Exceedances (Section 6Av):** MS/MSD relative percent differences (RPDs) were outside the acceptable limits as listed below. Results did not require qualification.

| Compound | MS/MSD RPD | RPD Limits | Results Qualified |
|---|---|---|---|
| **EP21-WAT-BULK-03-071822:** | | | |
| NFDHA | 32.8 | 30 | The NFDHA results for samples EP21-WAT-BULK-01-071822 / EP21-WAT-BULK-02-071822 / EP21-WAT-BULK-03-071822 were nondetect and did not require qualification due to the RPD exceedance. |

**Field Duplicates (Section 6Bi):** Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **EP21-WAT-BULK-01-071822 / EP21-WAT-BULK-02-071822 / EP21-WAT-BULK-03-071822:** | | | | |
| PFHpA | 6.33 | 4.77 | 4.66 | **31.8** |
| PFOA | 92.2 | 70.6 | 67.5 | **32.2** |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW10A-GW-273-283-01-071422 | L37502-61 | PFOS |
| MW10B-GW-130-140-01-071422 | L37502-62 | PFOS |
| MW10C-GW-10-20-01-071322 | L37502-63 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82860 (WG82620) Report date (mm/dd/yyyy): 10/7/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/27/2022

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | The samples were received at temperatures of 4.7, 7.1, and 9.7 °C, and some of the samples were above the ≤ 6 °C criteria. Results were not qualified as the samples were received at temperatures of ≤ 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER5-072022 (L37502-5) and TRIP-COOLER6-072022 (L37502-6) - reported in SDG DPWG82722 (WG82115)<br>TRIP-COOLER1-082222 (L37762-1)<br>EQ-DRILLERPUMP-01-081822 L37762-2<br>EQ-DRILLERPUMP-02-081822 (L37762-3)<br>EQ-DRILLWATER-01-081822 (L37762-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP21-WAT-BULK-03-071822 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field triplicate: EP21-WAT-BULK-01-071822 / EP21-WAT-BULK-02-071822 /EP21-WAT-BULK-03-071822 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit used for groundwater matrix<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes was used for ssample EP21-WAT-BULK-01-071822 (L37502-55) because the sample arrived with the cap cracked, and only half the sample volume remained. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (December 2021).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2022

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG82939 (WG82650)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/30/22 20:52 | NFDHA | 144% | 70-130% | The associated results were nondetect and were acceptable without qualification. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ). Labeled analog exceedances are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW26B-SED-COMP-30-35-01-072122 | L37707-1 | D5-N-EtFOSA | 49.7 | UJ |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG82939 (WG82650) Report date (mm/dd/yyyy): 10/21/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 1/22/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples (not reported in this SDG) were listed on the COCs. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | The associated trip blank was not reported in this SDG:<br>TRIP-COOLER2-081222 (L37706-2)<br>Reported in SDG DPWG83492 (WG83076) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☐ | ☒ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/22/2023

**SGS AXYS DPWG81620** 1/22/23 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | | | | | |
|---|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | | p 773 |
| | | | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 | | |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 | | |
| 27916 | 6263 | 2 | 1.1287 | 1.13 | | |
| 23878 | 13307 | 5 | 1.1215 | 1.11 | | |
| 24247 | 28296 | 10 | 1.1742 | 1.17 | | |
| 26274 | 63187 | 20 | 1.2099 | 1.21 | | |
| 26621 | 144547 | 50 | 1.0927 | 1.09 | | |
| 25106 | 683620 | 250 | 1.0959 | 1.09 | | |
| | | Avg: | 1.1373 | 1.13 | 1.13401 | (raw data) |
| | | %RSD: | 5.9989 | 6.15 | | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW27A-GW-221-222-01-071122 | PFOS | 358967 | 13776 | 1 | 418.09 | 418 | 0.5530 | 0.004 | L37706-3 |
| MW22C-GW-160-170-01-072522 | PFOS | 20697 | 19034 | 1 | 146.41 | 146 | 0.0659 | 0.004 | L37706-9 |
| WG83076-102 | PFOS | 9842 | 20271 | 1 | 4.31 | 4.31 | 1.00 | 0.004 | |
| WG83076-104 | PFOS | 9604 | 18347 | 1 | 4.64 | 4.64 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 10/21/22 23:50 | 14334 | 26902 | 4.728 | 4.71 | 5.00 | 94.2 | 94.3% |
| CCV 10/22/22 3:33 | 13975 | 26282 | 4.718 | 4.72 | 5.00 | 94.4 | 94.4% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG83076-101): | PFOS | 0.020 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG83492 (Analysis WG83076) dated 11/23/2022 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 1/22/2022 |

The groundwater samples and associated trip blank samples listed below were collected at the Project 1007 Site on July 11 through September 7, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW27A-GW-221-222-01-071122 | | 7/11/2022 | L37706-3 |
| MW27A-GW-221-222-01-071822 | | 7/18/2022 | L37706-4 |
| MW27B-GW-120-130-01-071422 | | 7/14/2022 | L37706-5 |
| MW21A-GW-410-420-01-071422 | | 7/14/2022 | L37706-6 |
| MW22A-GW-429-439-01-071422 | | 7/14/2022 | L37706-7 |
| MW22B-GW-290-390-01-071422 | | 7/14/2022 | L37706-8 |
| MW22C-GW-160-170-01-072522 | | 7/25/2022 | L37706-9 |
| MW26A-GW-380-390-01-081022 | | 8/10/2022 | L37706-10 |
| MW26B-GW-31-35-01-072122 | | 7/21/2022 | L37706-11 |
| MW26B-GW-31-35-02-072122 | Field Duplicate of MW26B-GW-31-35-01-072122 | 7/21/2022 | L37706-12 |
| MW26B-GW-249-259-01-080822 | | 8/8/2022 | L37706-13 |
| MW26C-GW-130-140-01-080822 | | 8/8/2022 | L37706-14 |
| MW8A-GW-166-170-01-081622 | | 8/16/2022 | L37706-15 |
| MW8A-GW-202-212-01-090722 | | 9/7/2022 | L37906-1 |
| MW8B-GW-50-60-01-090722 | | 9/7/2022 | L37906-2 |
| **Field QC Blanks:** | | | |
| TRIP-COOLER1-081222 | Trip Blank | 8/12/2022 | L37706-1 |
| TRIP-COOLER2-081222 | Trip Blank | 8/12/2022 | L37706-2 |

Data validation activities were conducted with reference to:

- Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2022.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

## REVIEW ELEMENTS

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification (CCVs) |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control samples (LCSs) |
| ✓ | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample receipt temperature, labeled compound recoveries, LCS recoveries, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

The sample was received at the laboratory intact; however, eight samples were above the 6 °C temperature requirement at 13.9 °C. These sample detects were qualified as estimated biased low (J-) and nondetect results were qualified UJ as the samples were received at a temperature greater than 10 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

## Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA (highest 2 standards), and PFUnA from the initial calibration curve due to non-linearity at the high concentrations.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point, and this calibration point was not used during calibration. In addition, the NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 10/22/22 6:23 | NFDHA | 144 | 70-130 | The associated results were nondetect and were acceptable without qualification. |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

## Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Two trip blanks were associated with the sample data. The trip blank results were nondetect.

## Labeled Compounds

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER2-081222 | L37706-2 | d9-NEt-FOSE | 49.0 | UJ |
| MW27A-GW-221-222-01-071122 | L37706-3 | 13C2-PFTeDA | 47.8 | UJ<br>PFTrDA: UJ |
| MW27A-GW-221-222-01-071122 | L37706-3 | D5-N-EtFOSA | 44.0 | UJ |
| MW27A-GW-221-222-01-071122 | L37706-3 | d9-NEt-FOSE | 48.0 | UJ |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD results were not included with this data set.

## LCS/LCSD Results

LCS/LCSDs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits, except as listed in bold in the table below.

| LCS IDs | Compound | LCS/LCSD % Recovery | Recovery Limits | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG83076-102<br>WG83076-104 | NFDHA | 92.1/**143** | 70-130 | **43.3** | 30 | NFDHA results were nondetect and were acceptable without qualification. |
| WG83076-103 (Low Level) | N-MeFOSA | **60.0**/-- | 70-130 | -- | -- | N-EtFOSA and N-EtFOSA results were nondetect and qualified as estimated (UJ). |
| | N-EtFOSA | **60.1**/-- | 70-130 | -- | -- | |

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Sample MW26B-GW-31-35-02-072122 was collected as a field duplicate of MW26B-GW-31-35-01-072122. RPDs were within the 30 percent limit for water sample, or results were within ± the limit of quantification.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

A smaller sample volume was used for sample prep for most samples. The sample prep logs indicated this was due to high solids content.

Quantitations were performed to include branched isomers where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and are considered biased high (J+). For most samples, the PFOS qualification is attributable to an ion ratio exceedance of PFOS-IV which is present at a low concentration, but does not meet ion ratio criteria. The PFOS qualifier was updated to estimated (J) for samples received at an elevated temperature, as both high bias and low bias are present.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW27A-GW-221-222-01-071122 | L37706-3 | PFOS | No |
| MW27A-GW-221-222-01-071822 | L37706-4 | PFOS | No |
| MW21A-GW-410-420-01-071422 | L37706-6 | PFOS | No |
| MW22A-GW-429-439-01-071422 | L37706-7 | PFOS | No |
| MW22B-GW-290-390-01-071422 | L37706-8 | PFOS | No |
| MW22C-GW-160-170-01-072522 | L37706-9 | PFOS | No |
| MW26A-GW-380-390-01-081022 | L37706-10 | PFOS | Yes |
| MW26B-GW-249-259-01-080822 | L37706-13 | PFOS | No |

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW8A-GW-166-170-01-081622 | L37706-15 | PFHxS | No |
| MW8A-GW-166-170-01-081622 | L37706-15 | PFOS | No |
| MW8A-GW-202-212-01-090722 | L37906-1 | PFOS | No |
| MW8B-GW-50-60-01-090722 | L37906-2 | PFHxS | No |
| MW8B-GW-50-60-01-090722 | L37906-2 | PFOS | No |

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW27A-GW-221-222-01-071122 (L37706-3)<br>MW27A-GW-221-222-01-071822 (L37706-4)<br>MW27B-GW-120-130-01-071422 (L37706-5)<br>MW21A-GW-410-420-01-071422 (L37706-6)<br>MW22A-GW-429-439-01-071422 (L37706-7)<br>MW22B-GW-290-390-01-071422 (L37706-8)<br>TRIP-COOLER1-081222 (L37706-1)<br>TRIP-COOLER2-081222 (L37706-2) | All PFAS | ng/L | Detects: J-<br>Nondetects: UJ | sr |
| MW27A-GW-221-222-01-071122 (L37706-3)<br>TRIP-COOLER2-081222 (L37706-2) | N-EtFOSE | ng/L | UJ | s |
| MW27A-GW-221-222-01-071122 (L37706-3) | PFTeDA<br>PFTrDA<br>N-EtFOSA | ng/L | UJ | s |
| All samples | N-MeFOSA<br>N-EtFOSA | ng/L | UJ | l |
| MW26A-GW-380-390-01-081022 (L37706-10) | PFOS | ng/L | J+ | em |
| MW27A-GW-221-222-01-071122 (L37706-3)<br>MW27A-GW-221-222-01-071822 (L37706-4)<br>MW21A-GW-410-420-01-071422 (L37706-6)<br>MW22A-GW-429-439-01-071422 (L37706-7)<br>MW22B-GW-290-390-01-071422 (L37706-8) | PFOS | ng/L | J | q |
| MW22C-GW-160-170-01-072522 (L37706-9)<br>MW26B-GW-249-259-01-080822 (L37706-13)<br>MW8A-GW-166-170-01-081622 (L37706-15)<br>MW8A-GW-202-212-01-090722 (L37906-1)<br>MW8B-GW-50-60-01-090722 (L37906-2) | PFOS | ng/L | J+ | q |
| MW8A-GW-166-170-01-081622 (L37706-15)<br>MW8B-GW-50-60-01-090722 (L37906-2) | PFHxS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

em Estimated maximum concentration
l Laboratory control sample
q Quantitation
s Labeled compound

**Attachment A**

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG84008 (WG83803)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at a temperature of 9.6 °C. Results were not qualified, as they were received at a temperature < 10 °C.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/6/23 21:53 | NFDHA | 206% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 1/7/23 2:03 | NFDHA | 224% | 70-130% | |

**Blank detections (Section 3Aii):** Equipment blank detections and the results qualified are listed in the table below.

| Blank Sample ID | Analyte | Concentration (ng/L) | Qualifications |
|---|---|---|---|
| EQ-BOTTLE-01-092022 | PFOS | 3.14 | Associated detects that were within ten times the blank concentration were qualified B:<br><br>MW2A-GW-240-263-01-091922 (L38126-3)<br>PZH-3-GW-36-40-01-092022 (L38126-8) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2A-GW-240-263-01-091922 | L38126-3 | 13C4-PFBA | 13.0 | J+ |
| MW2A-GW-240-263-01-091922 | L38126-3 | 13C2-4:2 FTS | 151 | No Qualifier |
| PZH-2-GW-36-40-01-092022 | L38126-6 | 13C7-PFUnA | 35.4 | UJ |
| PZH-2-GW-36-40-01-092022 | L38126-6 | D5-N-EtFOSAA | 42.0 | UJ |
| PZH-2-GW-36-40-01-092022 | L38126-6 | 13C2-PFDoA | 13.0 | UJ |
| PZH-2-GW-36-40-01-092022 | L38126-6 | D5-N-EtFOSA | 14.2 | UJ |
| PZH-2-GW-36-40-01-092022 | L38126-6 | D3-N-MeFOSA | 22.7 | UJ |
| PZH-2-GW-36-40-01-092022 | L38126-6 | 13C8-PFOS | 15.3 | UJ |
| PZH-2-GW-46-50-01-092022 | L38126-7 | d9-NEt-FOSE | 45.0 | UJ |
| PZH-2-GW-46-50-01-092022 | L38126-7 | 13C2-PFTeDA | 44.0 | UJ<br>PFTrDA: UJ |

Four results for sample PZH-2-GW-36-40-01-092022 (L38126-6) were reported by the laboratory as not quantifiable (NQ), as the labeled analog recoveries were less than 1%. A validation qualifier of rejected (R) was used for these results, as a result was not reported.

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|---|---|
| WG83803-102 WG83803-104 (DUP WG83803-102) | 3:3 FTCA | 77.9/**33.7** | 70-130 | **79.1** | 30 | The associated results were nondetect and qualified as estimated (UJ). |
| | PFMPA | 80.8/**42.8** | 70-130 | **61.6** | 30 | Detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |
| | 11Cl-PF3OUdS | 84.3/122 | 70-130 | **36.6** | 30 | Associated results were nondetect, and were acceptable without qualification. |
| | 4:2 FTS | **153**/114 | 70-130 | 29.4 | 30 | |
| | 9Cl-PF3ONS | 90.0/129 | 70-130 | **36.0** | 30 | |
| | NFDHA | **209/231** | 70-130 | 9.98 | 30 | |
| WG83803-103 (Low Level) | PFHpA | **151**/-- | 70-130 | -- | -- | Detects were qualified as estimated biased high (J+). |
| | 4:2 FTS | **146**/-- | 70-130 | -- | -- | Associated results were nondetect, and were acceptable without qualification. |
| | 6:2 FTS | **161**/-- | 70-130 | -- | -- | |
| | NFDHA | **231**/-- | 70-130 | -- | -- | |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZH-3-GW-36-40-01-092022 | L38126-8 | PFHxA |

The PFOS result for sample MW2F-GW-130-150-01-093022 (L38126-5) was reported from a five times dilution analysis due to the high concentration.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG84008 (WG83803) Report date (mm/dd/yyyy): 1/18/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 2/27/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | . |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 9.6 °C, and above the ≤ 6 °C criteria. Results did not require qualification as the samples were received at a temperature of less than 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-100522<br>EQ-BOTTLE-01-092022 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | EQ-BOTTLE-01-092022:<br>PFOS 3.14 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | Four results for sample PZH-2-GW-36-40-01-092022 (L38126-6) were reported by the laboratory as not quantifiable (NQ), as the labeled analog recoveries were less than 1%. A validation qualifier of rejected (R) was used for these results, as a result was not reported. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit used for water matrix.<br>See data qualifications summary (attachment) for exceedances. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Four results were not reported for sample PZH-2-GW-36-40-01-092022 (L38126-6) due to severely low labeled analog compound recoveries. These results were qualified as rejected (R).**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 2/27/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85019 (WG84072)**

**Chain of custody preservation, and holding time (Section 1C):** Most samples were received above the < 6 °C criteria at temperatures of 7.3 °C, 7.4 °C, 8.1 °C and 8.9 °C. Results were not qualified, as they were received at a temperature < 10 °C.

Samples MW21A-GW-410-420-01-102722 (L38327-8), MW15A-GW-330-340-01-102722 (L38327-9), MW6A-GW-185-192-01-102722 (L38327-10), and MW6B-GW-140-150-01-102722 (L38327-16) were extracted one day over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/1/23 21:51 | NFDHA | 295% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/2/23 1:48 | NFDHA | 275% | 70-130% | |
| CCV 2/2/23 4:38 | NFDHA | 189% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW21A-GW-410-420-01-102722 | L38327-8 | 13C2-4:2 FTS | 240 | No Qualifier |
| MW6A-GW-185-192-01-102722 | L38327-10 | 13C4-PFBA | 17.6 | J+ |
| MW6A-GW-185-192-01-102722 | L38327-10 | 13C2-4:2 FTS | 197 | No Qualifier |
| MW6B-GW-140-150-01-102722 | L38327-16 | 13C4-PFBA | 10.1 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG84072-102 | NFDHA | **249** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |
| WG84072-103 (Low Level) | 5:3 FTCA | **136** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |
| | NFDHA | **340** | 70-130 | |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW6B-GW-140-150-01-102722 | L38327-16 | PFBS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85019 (WG84072) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/14/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Most samples were received above the < 6 °C criteria at temperatures of 7.3 °C, 7.4 °C, 8.1 °C and 8.9 °C. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature and hold time exceedances are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-110222 (L38327-1)<br>TRIP-COOLER2-110222 (L38327-2)<br>TRIP-COOLER3-110222 (L38327-3)<br>TRIP-COOLER4-110222 (L38327-4)<br>TRIP-COOLER5-110222 (L38327-5)<br>TRIP-COOLER6-110222 (L38327-6)<br>TRIP-COOLER7-110222 (L38327-7) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None in this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | None in this SDG. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/14/2023

**SGS AXYS DPWG81620** 7/1/23 **LS**

Initial Cal 4/11/22 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 29658 | 1465 | 0.4 | 1.2426 | 1.24 |
| 26149 | 2148 | 0.8 | 1.0332 | 1.03 |
| 27916 | 6263 | 2 | 1.1287 | 1.13 |
| 23878 | 13307 | 5 | 1.1215 | 1.11 |
| 24247 | 28296 | 10 | 1.1742 | 1.17 |
| 26274 | 63187 | 20 | 1.2099 | 1.21 |
| 26621 | 144547 | 50 | 1.0927 | 1.09 |
| 25106 | 683620 | 250 | 1.0959 | 1.09 |
| | | Avg: | 1.1373 | 1.13 |
| | | %RSD: | 5.9989 | 6.15 |

p 773

1.13401 (raw data)

Internal Std Conc: 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| OW7Q-1-GW-64-74-01-102722 | PFOS | 32212 | 12813 | 1 | 45.25 | 45.3 | 0.4930 | 0.004 | L38327-11 |
| OW7S-1-GW-90-100-01-102722 | PFOS | 9394 | 19669 | 1 | 8.39 | 8.39 | 0.5050 | 0.004 | L38327-14 |
| WG84072-102 | PFOS | 11263 | 20800 | 1 | 4.80 | 4.80 | 1.00 | 0.004 | |
| WG84072-103 | PFOS | 3681 | 20353 | 1 | 1.60 | 1.60 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 2/2/23 1:48 | 10892 | 21569 | 4.481 | 4.48 | 5.00 | 89.6 | 89.6% |
| CCV 2/2/23 4:38 | 11579 | 21010 | 4.890 | 4.87 | 5.00 | 97.4 | 97.4% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG84072-101): | PFOS | 0.008 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG85020 (Analysis WG84072) dated 7/6/2023 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 7/19/2023 |

The groundwater samples and associated trip blank samples listed below were collected at the Project 1007 Site on October 27, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| OW7Q-1-GW-64-74-01-102722 | | 10/27/2022 | L38327-11 |
| OW7Q-1-GW-64-74-02-102722 | Field Duplicate of OW7Q-1-GW-64-74-01-102722 | 10/27/2022 | L38327-12 |
| OW7Q-1-GW-64-74-03-102722 | Field Triplicate of OW7Q-1-GW-64-74-01-102722; MS/MSD | 10/27/2022 | L38327-13 (A) |
| OW7S-1-GW-90-100-01-102722 | | 10/27/2022 | L38327-14 |
| PW7S-1-GW-100-150-01-102722 | | 10/27/2022 | L38327-15 |

*Data validation activities were conducted with reference to:*

- *Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2022.*
- *Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.*

*Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).*

***REVIEW ELEMENTS***

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✗ Sample Receipt and Holding times
✓ Initial calibration/continuing calibration verification (CCVs)
✓ Laboratory and trip blanks
✗ Labeled Compounds
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓ Laboratory control samples (LCSs)

✓ Field duplicates

✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to sample hold times, labeled compound recoveries, MS/MSDs, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

A resubmitted data package dated 7/6/2023 was received that included corrected chain of custody documentation and confirmation of the holding time exceedances. The resubmitted analytical data package was verified as complete.

### Sample Receipt and Holding Times

The sample was received at the laboratory intact; however, the samples were above the 6 °C temperature requirement at 7.4 °C. Results were not qualified as the samples were received at a temperature greater than 10 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted one day over the 90-day hold time and detects were qualified as estimated biased low (J-) and nondetects were qualified UJ.

### Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%. The highest calibration standard was excluded for EtFOSAA, MeFOSAA, NFDHA (highest 2 standards), and PFUnA from the initial calibration curve due to non-linearity at the high concentrations.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 164% for the second highest (100) calibration point, and this calibration point was not used during calibration. In addition, the NFDHA sample results were nondetect, and were not affected by this deviation.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation.

Second source verification recoveries were within ± 30% of the true value, with the exception of NFDHA which reported a recovery of 145%. NFDHA results were nondetect and were acceptable without qualification.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration | Analyte | %R | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/1/2023 21:51 | NFDHA | 295 | 70-130 | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/2/2023 1:48 | NFDHA | 275 | 70-130 | |
| CCV 2/2/2023 4:48 | NFDHA | 189 | 70-130 | |

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. One trip blank (TRIP-COOLER1-110222, L38327-1) was associated with the sample data; however, it was not reported in this SDG. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW7Q-1-GW-64-74-02-102722 | L38327-12 | 13C2-6:2 FTS | 152 | No Qualifier |
| OW7Q-1-GW-64-74-02-102722 | L38327-12 | 13C2-PFTeDA | 47.6 | UJ<br>PFTrDA: UJ |
| OW7Q-1-GW-64-74-03-102722 | L38327-13 (A) | 13C2-6:2 FTS | 155 | No Qualifier |
| OW7S-1-GW-90-100-01-102722 | L38327-14 | 13C2-6:2 FTS | 300 | No Qualifier |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD OW7Q-1-GW-64-74-03-102722 was associated with samples OW7Q-1-GW-64-74-01-102722 and OW7Q-1-GW-64-74-02-102722, and recoveries and relative percent differences (RPDs) were with MPCA limits, except as summarized below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **OW7Q-1-GW-64-74-03-102722:** | | | | | |
| PFHxA | **169 /** 95.7 | 70-130 | 27.2 | 30 | The results for samples OW7Q-1-GW-64-74- |

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| PFHpA | **131** / 97.3 | 70-130 | 18.4 | 30 | 03-102722, OW7Q-1-GW-64-74-01-102722 and OW7Q-1-GW-64-74-02-102722 were qualified as estimated (J), as low bias also exist (hold times). |
| PFBS | **140** / 70.7 | 70-130 | **49.1** | 30 | |
| 6:2 FTS | **143** / 112 | 70-130 | 24.0 | 30 | The result for samples OW7Q-1-GW-64-74-03-102722, OW7Q-1-GW-64-74-01-102722 and OW7Q-1-GW-64-74-02-102722 were nondetect and were acceptable without qualification. |
| NFDHA | **372** / **192** | 70-130 | **64.2** | 30 | |
| PFBA | **166** / 110 | 70-130 | 5.13 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

## LCS Results

LCS are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits, except as listed in bold in the table below.

| LCS IDs | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG84072-102 | NFDHA | 249 | 70-130 | NFDHA results were nondetect and were acceptable without qualification. |
| WG84072-103 (Low Level) | 5:3 FTCA | 136 | 70-130 | 5:3 FTCA and NFDHA results were nondetect and were acceptable without qualification. |
| | NFDHA | 340 | 70-130 | |

## Field Duplicate Results

Samples OW7Q-1-GW-64-74-02-102722 and OW7Q-1-GW-64-74-03-102722 were collected to assess for field precision for sample OW7Q-1-GW-64-74-01-102722. RPDs were within the 30 percent limit for water sample, or results were within ± the reporting limit (RL). RPDs were within the 30 percent limit for water sample, or results were within ± the limit of quantification.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Quantitations were performed to include branched isomers where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1; and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and are considered biased high (J+). For most samples, the PFOS qualification is attributable to an ion ratio exceedance of PFOS-IV which is present at a low concentration, but does not meet ion ratio criteria. The PFOS qualifiers were updated to estimated (J) for samples with ion ratio exceedances (high bias) and hold time exceedances (low bias) as both high and low bias are present.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| OW7Q-1-GW-64-74-01-102722 | L38327-11 | PFOS | No |
| OW7Q-1-GW-64-74-02-102722 | L38327-12 | PFOS | No |
| OW7Q-1-GW-64-74-03-102722 | L38327-13 (A) | PFOS | No |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high and low bias were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| OW7Q-1-GW-64-74-01-102722 (L38327-11)<br>OW7Q-1-GW-64-74-02-102722 (L38327-12)<br>OW7Q-1-GW-64-74-03-102722 (L38327-13 (A))<br>OW7S-1-GW-90-100-01-102722 (L38327-14)<br>PW7S-1-GW-100-150-01-102722 (L38327-15) | All PFAS | ng/L | Detects: J-<br>Nondetects: UJ | ht |
| OW7Q-1-GW-64-74-02-102722 (L38327-12) | PFTeDA<br>PFTrDA | ng/L | UJ | s |
| OW7Q-1-GW-64-74-01-102722 (L38327-11)<br>OW7Q-1-GW-64-74-02-102722 (L38327-12)<br>OW7Q-1-GW-64-74-03-102722 (L38327-13 (A)) | PFHxA<br>PFHpA<br>PFBS | ng/L | J | ms |
| OW7Q-1-GW-64-74-01-102722 (L38327-11)<br>OW7Q-1-GW-64-74-02-102722 (L38327-12)<br>OW7Q-1-GW-64-74-03-102722 (L38327-13 (A)) | PFOS | ng/L | J | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

ht Hold time
ms Matrix spike
q Quantitation
s Labeled compound

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85479 (WG84073)**

**Chain of custody preservation, and holding time (Section 1C and 1E):** Samples were received at temperatures of 7.3°C, 8.1°C, and 8.9°C. Results were not qualified, as they were received at a temperature < 10 °C.

Samples were extracted 5 to 6 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/6/23 23:47 | NFDHA | 381% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/7/23 2:51 | NFDHA | 283% | 70-130% | |
| CCV 2/7/23 5:02 | PFHpA | 131% | 70-130% | Associated sample detects were qualified as estimated biased high (J+):<br><br>MW6C-GW-35-40-03-102722 (L38327-19 (A))<br>OW7S-2-GW-100-110-01-102722 (L38327-31)<br>OW7S-3-GW-100-110-01-102722 (L38327-32)<br>MW1A-GW-360-370-01-102722 (L38327-33)<br>MW2A-GW-240-263-01-102622 (L38327-34)<br>MW2D-GW-7-17-01-102622 (L38327-35)<br>MW22B-GW-290-300-01-102722 (L38327-37)<br>MW22C-GW-160-170-01-102722 (L38327-38) |
| | NFDHA | 249% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/7/23 8:06 | NFDHA | 334% | 70-130% | The associated results were nondetect and were acceptable without qualification. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW4A-GW-140-160-01-102822 | L38327-24 | 13C2-4:2 FTS | 153 | No Qualifier |
| MW2C-GW-35-40-01-102822 | L38327-25 | 13C3-HFPO-DA | 31.7 | UJ |
| MW2C-GW-35-40-01-102822 | L38327-25 | 13C2-4:2 FTS | 157 | No Qualifier |
| MW9A-GW-140-150-01-102722 | L38327-27 | 13C4-PFBA | 4.04 | R |
| MW9A-GW-140-150-01-102722 | L38327-27 | 13C2-8:2 FTS | 44.7 | UJ |
| OW7S-2-GW-100-110-01-102722 | L38327-31 | 13C4-PFBA | 26.0 | J+ |
| OW7S-3-GW-100-110-01-102722 | L38327-32 | 13C2-4:2 FTS | 181 | No Qualifier |
| MW1A-GW-360-370-01-102722 | L38327-33 | 13C4-PFBA | 3.75 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW1A-GW-360-370-01-102722 | L38327-33 | 13C2-4:2 FTS | 155 | No Qualifier |
| MW2A-GW-240-263-01-102622 | L38327-34 | 13C4-PFBA | 25.4 | J+ |
| MW22A-GW-429-439-01-102722 | L38327-36 | 13C4-PFBA | 22.6 | J+ |
| MW22B-GW-290-300-01-102722 | L38327-37 | 13C4-PFBA | 40.7 | J+ |
| MW22B-GW-290-300-01-102722 | L38327-37 | 13C2-4:2 FTS | 167 | No Qualifier |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG84073-102 | NFDHA | **240** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |
| WG84073-103 (Low Level) | NFDHA | **243** | 30-130 | |

**Bold** indicates an exceedance

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW6C-GW-35-40-03-102722:** | | | | | |
| PFHxA | 113 / **47.6** | 70-130 | **30.2** | 30 | The results for samples MW6C-GW-35-40-01-102722, MW6C-GW-35-40-02-102722, and MW6C-GW-35-40-03-102722 were qualified as estimated biased low (J-). |
| NFDHA | **253 / 256** | 70-130 | 0.653 | 30 | The results for samples MW6C-GW-35-40-01-102722, MW6C-GW-35-40-02-102722, and MW6C-GW-35-40-03-102722 were nondetect, and were acceptable without qualification. |
| PFBA | **257 / 205** | 70-130 | 2.69 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **51.7** / 93.0 | 70-130 | 5.95 | 30 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW9A-GW-140-150-01-102722 | L38327-27 | PFHxA |
| MW13C-GW-115-125-01-102822 | L38327-28 | PFOS |
| MW13D-GW-15-25-01-102822 | L38327-29 | PFOS |
| OW7S-2-GW-100-110-01-102722 | L38327-31 | PFNA |
| OW7S-3-GW-100-110-01-102722 | L38327-32 | PFDA |
| MW1A-GW-360-370-01-102722 | L38327-33 | PFOS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW2A-GW-240-263-01-102622 | L38327-34 | PFOS |
| MW2D-GW-7-17-01-102622 | L38327-35 | PFHxA |

The PFOS result for samples MW2C-GW-35-40-02-102822 (L38327-26), MW14D-GW-6-21-01-102822 (L38327-30), and MW22A-GW-429-439-01-102722 (L38327-36) were reported from five times dilution analyses due to the high concentration.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85479 (WG84073) Report date (mm/dd/yyyy): 7/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/14/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | .The COC for cooler 2 listed the sampe matrix for only the first sample. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at temperatures of 7.3°C, 8.1°C, and 8.9°C, and above the ≤ 6 °C criteria. Results did not require qualification as the samples were received at a temperature of less than 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER2-110222 (L38327-2)<br>TRIP-COOLER3-110222 (L38327-3)<br>TRIP-COOLER4-110222 (L38327-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW6C-GW-35-40-03-102722 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>Field triplicate: MW6C-GW-35-40-01-102722 /<br>MW6C-GW-35-40-02-102722 / |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | MW6C-GW-35-40-03-102722<br>Field Duplicate: MW2C-GW-35-40-01-102822 / MW2C-GW-35-40-02-102822 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit used for water matrix.<br>RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Two PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/14/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85533 (WG84133)**

**Chain of custody preservation, and holding time (Section 1C):** Nine of the samples were received at a temperature of 8.4°C and three samples were received at a temperature of 7.8 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C.

Samples were extracted five to twelve days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/14/23 10:40 | NFDHA | 325% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/14/23 14:10 | NFDHA | 197% | 70-130% | |
| CCV 2/14/23 18:23 | 11Cl-PF3OUdS | 141% | 70-130% | |
| | NFDHA | 340% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ), and detections associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Nondetect results associated with high recoveries were acceptable without qualification. Four PFBA results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BL5-WAT-BULK-02-102522 | L38327-59 | 13C9-PFNA | 181 | J- |
| OD3-WAT-BULK-01-102522 | L38327-60 | 13C2-4:2 FTS | 162 | No Qualifier |
| MW15B-GW-215-225-01-102722 | L38327-69 | 13C2-8:2 FTS | 38.0 | UJ |
| MW15B-GW-215-225-01-102722 | L38327-69 | D5-EtFOSAA | 47.7 | UJ |
| MW15B-GW-215-225-01-102722 | L38327-69 | D3-MeFOSAA | 46.9 | UJ |
| MW15B-GW-215-225-01-102722 | L38327-69 | 13C4-PFBA | 1.15 | R |
| MW15B-GW-215-225-01-102722 | L38327-69 | 13C5-PFPeA | 23.4 | UJ |
| MW9B-GW-90-100-01-102722 | L38327-70 | 13C4-PFBA | 8.25 | R |
| OW5J-1-GW-230-240-01-103122 | L38375-58 | 13C2-4:2 FTS | 153 | No Qualifier |
| OW5J-1-GW-230-240-01-103122 | L38375-58 | 13C4-PFBA | 6.22 | R |
| OW5O-1-GW-200-210-01-103122 | L38375-59 | 13C4-PFBA | 4.74 | R |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-102522:** | | | | | |
| PFBA | **64.1 /** 114 | 70-130 | 14.6 | 30 | The results for samples RC5-WAT-BULK-03-102522, RC5-WAT-BULK-03-102522, and RC5-WAT-BULK-03-102522 were qualified as estimated biased low (J-). |
| PFHxA | **153 / 64.7** | 70-130 | **34.1** | 30 | The results for samples RC5-WAT-BULK-03-102522, RC5-WAT-BULK-03-102522, and RC5-WAT-BULK-03-102522 were qualified as estimated (J). |
| 5:3 FTCA | **139 /** 117 | 70-130 | 14.7 | 30 | The results for samples RC5-WAT-BULK-03-102522, RC5-WAT-BULK-03-102522, and RC5-WAT-BULK-03-102522 were nondetect and were acceptable without qualification. |
| 6:2 FTS | 112 **/132** | 70-130 | 9.95 | 30 | |
| HFPO-DA | 98.9 / **175** | 70-130 | **58.1** | 30 | |
| ADONA | 95.6 / **177** | 70-130 | **62.5** | 30 | |
| 11Cl-PF3OUdS | 83.4 / **154** | 70-130 | **62.0** | 30 | |
| NFDHA | **289 / 307** | 70-130 | 8.96 | 30 | |
| PFOA | **Not Reported / 194** | 70-130 | 29.7 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **305 / 438** | 70-130 | 5.23 | 30 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **RC5-WAT-BULK-01-102522 / RC5-WAT-BULK-02-102522 / RC5-WAT-BULK-03-102522:** | | | | |
| PFHpA | 32 | 45.5 | 39.1 | 34.7 |
| PFBS | 19.9 | 24.2 | 27.4 | 31.5 |
| **BL5-WAT-BULK-01-102522 / BL5-WAT-BULK-02-102522:** | | | | |
| PFHxA | 31.7 | 20.1 | -- | 44.8 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| BL5-WAT-BULK-01-102522 | L38327-58 | N-MeFOSAA |
| PZAD-GW-21-26-01-102622 | L38327-64 | PFOS |
| MW8A-GW-202-212-01-102722 | L38327-68 | PFNA |

Four samples were prepped using reduced sample volumes due to high analyte concentrations This resulted in higher reporting limits.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85533 (WG84133) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/19/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Nine of the samples were received at a temperature of 8.4°C and three samples were received at a temperature of 7.8 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER6-110222 (L38327-6)<br>TRIP-COOLER7-110222 (L38327-7)<br>TRIP-COOLER6-110722 (L38375-6) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-102522 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field triplicate: RC5-WAT-BULK-01-102522 /<br>RC5-WAT-BULK-02-102522 / |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | RC5-WAT-BULK-03-102522<br>Field Duplicate: BL5-WAT-BULK-01-102522 / BL5-WAT-BULK-02-102522 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± QL, used for water matrix.<br>See data qualifications summary (attachment) for MS/MSD RPD and FD/FT exceedances. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Four samples were prepped using reduced sample volumes due to high analyte concentrations This resulted in higher reporting limits. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Four PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/19/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85536 (WG84132)**

**Chain of custody preservation, and holding time (Section 1C):** Four samples were received at a temperature of 8.1 °C, one sample at 8.4 °C and three samples at 8.9 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C.

Samples were extracted eight to ten days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/10/23 00:26 | 4:2 FTS | 139% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| | NFDHA | 241% | 70-130% | |
| CCV 2/10/23 3:16 | ADONA | 138% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| | 9Cl-PF3ONS | 132% | 70-130% | |
| | 11Cl-PF3OUdS | 141% | 70-130% | |
| | NFDHA | 185% | 70-130% | |
| CCV 2/10/23 5:28 | NFDHA | 231% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/10/23 8:58 (closing) | 3:3 FTCA | 68.6% | 70-130% | Associated results were nondetect and qualified as estimated (UJ):<br><br>PZGD-GW-35-40-03-102522 {L38327-23 (A))<br>PZFD-GW-33-38-01-102522 {L38327-48)<br>PZFS-GW-18-28-01-102522 {L38327-49)<br>PZED-GW-16-21-01-102522 {L38327-50) |
| | NFDHA | 332% | 70-130% | The associated results were nondetect and were acceptable without qualification. |
| CCV 2/21/23 21:55 | NFDHA | 135% | 70-130% | This run was associated with PFHxA analysis only. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetect and were acceptable without qualification. One PFBA result was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZGD-GW-35-40-01-102522 | L38327-21 | 13C2-4:2 FT | 155 | No Qualifier |
| PZGD-GW-35-40-01-102522 | L38327-21 | 13C3-HFPO-DA | 49.5 | UJ |
| MW26A-GW-380-390-01-102722 | L38327-39 | 13C4-PFBA | 23.7 | J+ |
| MW27B-GW-120-130-01-102722 | L38327-41 | 13C4-PFBA | 7.43 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZID-GW-22-27-01-102622 | L38327-42 | 13C2-PFTeDA | 49.3 | UJ<br>PFTrDA: UJ |
| PZFS-GW-18-28-01-102522 | L38327-49 | 13C3-HFPO-DA | 49.9 | UJ |
| PZBD-GW-16-21-01-102622 | L38327-51 | 13C2-4:2 FT | 153 | No Qualifier |
| RC3-WAT-BULK-01-102522 | L38327-53 | 13C4-PFBA | 16.6 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG84132-102 | 6:2 FTS | **135** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |
| | NFDHA | **189** | 70-130 | |
| WG84132-103 (Low Level) | HFPO-DA | **67.0** | 70-130 | Associated HFPO-DA results were nondetect and qualified as estimated (UJ). |
| | NFDHA | **181** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |

**Bold** indicates an exceedance

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **PZGD-GW-35-40-03-102522:** | | | | | |
| PFOS | **454 / 423** | 70-130 | 5.91 | 30 | The results for samples PZGD-GW-35-40-01-102522, PZGD-GW-35-40-02-102522, and PZGD-GW-35-40-03-102522 were qualified as estimated biased high (J+). |
| PFHxA | **212 / 173** | 70-130 | 13.0 | 30 | |
| PFHpA | **140** / 81.3 | 70-130 | **43.9** | 30 | |
| 5:3 FTCA | **164 / 149** | 70-130 | 8.84 | 30 | The results for samples PZGD-GW-35-40-01-102522, PZGD-GW-35-40-02-102522, and PZGD-GW-35-40-03-102522 were nondetect and were acceptable without qualification. |
| 7:3 FTCA | **139 / 133** | 70-130 | 2.77 | 30 | |
| HFPO-DA | **136 /** 96.4 | 70-130 | **33.3** | 30 | |
| ADONA | **153 /** 124 | 70-130 | 20.3 | 30 | |
| 9Cl-PF3ONS | **135 /** 115 | 70-130 | 15.4 | 30 | |
| NFDHA | **336 / 273** | 70-130 | 19.5 | 30 | |
| PFEESA | **142 /** 127 | 70-130 | 10.2 | 30 | |
| PFBA | **143 / 131** | 70-130 | 1.14 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **PZGD-GW-35-40-01-102522 / PZGD-GW-35-40-02-102522 / PZGD-GW-35-40-03-102522:** | | | | |
| PFBS | 4.91 | 7.0 | 3.8 | 61.1 |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZGD-GW-35-40-01-102522 | L38327-21 | PFOS |
| PZGD-GW-35-40-02-102522 | L38327-22 | PFOS |
| PZGD-GW-35-40-03-102522 | L38327-23 (A) | PFOS |
| MW26A-GW-380-390-01-102722 | L38327-39 | PFOS |
| MW26A-GW-380-390-01-102722 | L38327-39 | PFNA |
| MW26B-GW-249-259-01-102722 | L38327-40 | PFOS |
| MW27B-GW-120-130-01-102722 | L38327-41 | PFOS |
| PZFD-GW-33-38-01-102522 | L38327-48 | PFHxA |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for sample PZFS-GW-18-28-01-102522 {L38327-49) was reported from a 5 times dilution analysis due to a high sample concentration. The PFHxA result for sample PZCS-GW-6-11-01-102622 {L38327-47) was reported from a reanalysis run due to instrument carry over during the initial analysis.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85536 (WG84132) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/14/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | The COC for cooler 2 listed the sampe matrix for only the first sample. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Four samples were received at a temperature of 8.1 °C, one sample at 8.4 °C and three samples at 8.9 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER2-110222 (L38327-2)<br>TRIP-COOLER4-110222 (L38327-4)<br>TRIP-COOLER5-110222 (L38327-5)<br>TRIP-COOLER6-110222 (L38327-6) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZGD-GW-35-40-03-102522 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD<br>Field triplicate:<br>PZGD-GW-35-40-01-102522<br>PZGD-GW-35-40-02-102522<br>PZGD-GW-35-40-03-102522<br>Field Duplicate:<br>PZIS-GW-7-17-01-102622<br>PZIS-GW-7-17-02-102622 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± QL, used for water matrix.<br>See data qualifications summary (attachment) for MS/MSD RPD and FT exceedances. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One PFBA result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/14/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85614 (WG84137)**

**Chain of custody preservation, and holding time (Section 1C):** Nine of the samples were received at a temperature of 7.2°C, and above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C.

Samples were extracted 25 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 4/18/23 11:30 | NFDHA | 146% | 70-130% | NFDHA results were not reported from this run (only a PFOS dilution was reported from this run). No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP21-WAT-BULK-01-102422 | L38375-36 | 13C4-PFBA | 18.0 | J+ |
| EP21-WAT-BULK-03-102422 | L38375-37 (A) | 13C4-PFBA | 38.8 | J+ |
| EP21-WAT-BULK-03-102422 | L38375-37 (A) | 13C2-PFTeDA | 41.8 | UJ |
| EP21-WAT-BULK-02-102422 | L38375-38 | 13C4-PFBA | 22.9 | J+ |
| EP2A-WADE-WAT-BULK-01-102422 | L38375-39 | 13C4-PFBA | 28.0 | J+ |
| EP2A-WAT-BULK-01-102422 | L38375-40 | D3-N-MeFOSA | 48.8 | UJ |
| EP2A-WAT-BULK-01-102422 | L38375-40 | 13C4-PFBA | 21.9 | J+ |
| EP2A-WAT-BULK-01-102422 | L38375-40 | 13C2-PFTeDA | 19.4 | UJ |
| EP2A-WAT-BULK-01-102422 | L38375-40 | D5-N-EtFOSA | 40.4 | UJ |
| EP26A-WAT-BULK-01-102422 | L38375-41 | 13C4-PFBA | 22.3 | J+ |
| EP27I-WADE-WAT-BULK-01-102422 | L38375-45 | 13C4-PFBA | 30.9 | J+ |
| EP16-WAT-BULK-01-102422 | L38375-46 | 13C4-PFBA | 37.6 | J+ |
| EP24-WAT-BULK-01-102422 | L38375-48 N | 13C4-PFBA | 38.3 | J+ |
| EP18E-WADE-WAT-BULK-01-102422 | L38375-49 | 13C4-PFBA | 26.1 | J+ |
| EP18E-WAT-BULK-01-102422 | L38375-51 | 13C4-PFBA | 41.3 | J+ |
| EP19-WAT-BULK-01-102422 | L38375-52 | 13C4-PFBA | 34.3 | J+ |
| EP27A-WAT-BULK-01-102422 | L38375-53 | 13C4-PFBA | 19.6 | J+ |
| WL6-WAT-BULK-01-102422 | L38375-54 | 13C4-PFBA | 40.0 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **EP21-WAT-BULK-03-102422:** | | | | | |
| PFOS | **340 / 248** | 70-130 | 2.93 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL6-WAT-BULK-01-102422 | L38375-54 | PFDA |

The PFOS result for samples EP26A-WAT-BULK-01-102422 (L38375-41), EP26D-WAT-BULK-01-102422 (L38375-43), EP18E-WAT-BULK-01-102422 (L38375-51), EP19-WAT-BULK-01-102422 (L38375-52), and PFBA for EP24-WAT-BULK-01-102422 (L38375-48 N) were reported from five times dilution analyses due to the high concentration.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85614 (WG84137) Report date (mm/dd/yyyy): 7/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 7/19/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | . |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Nine of the samples were received at a temperature of 7.2°C, and above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER3-110722 (L38375-3)<br>TRIP-COOLER4-110722 (L38375-4)<br>TRIP-COOLER5-110722 (L38375-5) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP21-WAT-BULK-03-102422 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs - ok<br>Field triplicate: EP21-WAT-BULK-01-102422<br>EP21-WAT-BULK-02-102422<br>EP21-WAT-BULK-03-102422 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix.<br>RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples EP26D-WAT-BULK-01-102422 (L38375-43) and EP26D-WADE-WAT-BULK-01-102422 (L38375-44) were prepped using reduced sample volumes due to high analyte concentrations This resulted in higher reporting limits. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 7/19/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85616 (WG84136)**

**Chain of custody preservation, and holding time (Section 1C):** Eight samples were received at a temperature of 8.8 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C.

Samples were extracted 17 to 22 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 3/30/23 1:34 | NFDHA | 69.2% | 70-130% | NFDHA was not reported from this CCV. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. Five results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW14A-GW-316-326-01-102622 | L38375-21 | 13C4-PFBA | 7.23 | R |
| MW7A-GW-200-210-01-102722 | L38375-26 | 13C2-8:2 FTS | 49.3 | UJ |
| MW7A-GW-200-210-01-102722 | L38375-26 | 13C4-PFBA | 3.71 | R |
| MW10B-GW-130-140-01-103122 | L38375-28 | 13C4-PFBA | 3.06 | R |
| MW5A-GW-210-220-01-103122 | L38375-30 | 13C4-PFBA | 47.1 | J+ |
| MW13A-GW-350-370-01-103122 | L38375-32 | 13C5-PFPeA | 45.9 | UJ<br>PFMPA: UJ<br>PFMBA: UJ |
| MW13A-GW-350-370-01-103122 | L38375-32 i | 13C4-PFBA | 5.63 | R |
| MW13B-GW-295-305-01-103122 | L38375-33 | 13C4-PFBA | 8.12 | R |
| W6201-GW-109-124-01-102822 | L38375-34 | d9-NEt-FOSE | 47.7 | UJ |
| W6201-GW-109-124-01-102822 | L38375-34 | D5-EtFOSAA | 45.3 | J+ |
| W6201-GW-109-124-01-102822 | L38375-34 | D5-N-EtFOSA | 35.1 | UJ |
| W6201-GW-109-124-01-102822 | L38375-34 | D3-MeFOSAA | 45.4 | UJ |
| W6201-GW-109-124-01-102822 | L38375-34 | D3-N-MeFOSA | 38.0 | UJ |
| W6201-GW-109-124-01-102822 | L38375-34 | 13C2-PFTeDA | 41.0 | UJ<br>PFTrDA: UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW2B-GW-57-62-03-102622:** | | | | | |
| PFBA | **166 / 161** | 70-130 | 0.235 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFHpA | **147 /** 110 | 70-130 | 5.03 | 30 | |
| PFOA | **382 / 324** | 70-130 | 1.05 | 30 | |
| PFHxS | **141 / 136** | 70-130 | 0.457 | 30 | |
| PFOS | **531 / 191** | 70-130 | 4.09 | 30 | |
| PFBS | **140 /** 118 | 70-130 | 4.79 | 30 | The results for samples MW2B-GW-57-62-01-102622, MW2B-GW-57-62-02-102622, and MW2B-GW-57-62-03-102622 were qualified as estimated biased high (J+). |
| 11Cl-PF3OUdS | 72.8 **/ 68.5** | 70-130 | 4.24 | 30 | The results for samples MW2B-GW-57-62-01-102622, MW2B-GW-57-62-02-102622, and MW2B-GW-57-62-03-102622 were nondetect and qualified as estimated (UJ). |
| NFDHA | 109 **/ 131** | 70-130 | 20.1 | 30 | The associated results were nondetect. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW5B-GW-110-120-01-103122 | L38375-31 | PFOS |
| W6201-GW-109-124-01-102822 | L38375-34 | PFOS |
| W6201-GW-109-124-01-102822 | L38375-34 | N-EtFOSAA |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

PFOS results for three samples, PFBA results for two samples and PFHxA for one sample were reported from 5 times dilution analyses because the concentration in the undiluted analyses exceeded the instrument calibration range.

Sample MW17B-GW-90-100-01-103122 was re-analyzed for PFOS and sample W13A-GW-350-370-01-103122 was reanalyzed for PFBA to confirm possible instrumental carry-over. The re-analysis showed that the original analysis data was affected by carry over and the re-analysis results for PFOS and PFBS were reported.

The initial instrumental analysis result of the sample MW7A-GW-200-210-01-102722 (SGS AXYS ID: L38375-26) did not meet method specifications for PFOS. The sample extract was reanalyzed and method specifications were met. The PFOS result for this sample was reported from the reanalysis.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85616 (WG84136) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/2/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Eight samples were received at a temperature of 8.8 °C. These samples were received above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature and hold times are discussed in the narrative. |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER1-110722 (L38375-1)<br>TRIP-COOLER2-110722 (L38375-2)<br>TRIP-COOLER3-110722 (L38375-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2B-GW-57-62-03-102622 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MS/MSD - RPDs ok<br>Field triplicate:<br>MW2B-GW-57-62-01-102622 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | MW2B-GW-57-62-02-102622<br>MW2B-GW-57-62-03-102622<br>Field duplicate (parent results not reported in this SDG):<br>MW1C-GW-40-45-01-102622<br>MW1C-GW-40-45-02-102622 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was use for four samples due to high suspended solids. A reduced volume was used for one sample due to high analyte concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Five results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/2/2023

**SGS AXYS DPWG85618** 9/4/23 **LS**

Initial Cal 2/19/23 **PFOS**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | p 765 |
| | | | | | |
| 26900 | 1525 | 0.4 | 1.4261 | 1.43 | |
| 37502 | 3424 | 0.8 | 1.1483 | 1.15 | |
| 24441 | 5305 | 2 | 1.0920 | 1.09 | |
| 27848 | 14531 | 5 | 1.0501 | 1.05 | |
| 31415 | 35990 | 10 | 1.1527 | 1.15 | |
| 35425 | 86522 | 20 | 1.2288 | 1.23 | |
| 33752 | 183693 | 50 | 1.0952 | 1.10 | |
| 26633 | 750554 | 250 | 1.1342 | 1.13 | |
| | | Avg: | 1.1659 | 1.17 | 1.16597 (raw data) |
| | | %RSD: | 10.0963 | 10.20 | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW20A-GW-130-140-01-103122 | PFOS | 184455 | 22198 | 1 | 146.05 | 146 | 0.4910 | 0.004 | L38383-4 |
| MW20A-GW-130-140-01-111722 | PFOS | 176889 | 22995 | 1 | 144.00 | 144 | 0.4610 | 0.004 | L38535-4 |
| WG84138-102 | PFOS | 16503 | 27568 | 1 | 5.17 | 5.17 | 1.00 | 0.004 | |
| WG84138-104 | PFOS | 15666 | 27130 | 1 | 4.98 | 4.98 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 3/6/23 20:21 | 18576 | 33974 | 4.718 | 4.72 | 5.00 | 94.40 | 94.4% |
| CCV 4/15/23 15:54 | 12998 | 25746 | 4.357 | 4.35 | 5.00 | 87.00 | 86.9% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG84138-101) | PFOS | 0.043 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG85618 (Analysis WG84138) dated 5/24/2023 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 9/5/2023 |

The groundwater samples listed below were collected at the Project 1007 Site October 28 through November 21, 2022.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| PZH-1-GW-90-100-01-110222 | | 11/2/2022 | L38383-1 |
| PZH-2-GW-40-50-01-102822 | | 10/28/2022 | L38383-2 |
| PZH-3-GW-30-40-01-102822 | | 10/28/2022 | L38383-3 |
| MW20A-GW-130-140-01-103122 | | 10/31/2022 | L38383-4 |
| MW20B-GW-90-100-01-103122 | | 10/31/2022 | L38383-5 |
| OW20P-1-GW-150-160-01-112122 | | 11/21/2022 | L38535-1 |
| PW20S-1-GW-200-250-01-112122 | | 11/21/2022 | L38535-2 |
| OW20T-1-GW-98-108-01-112122 | | 11/21/2022 | L38535-3 |
| MW20A-GW-130-140-01-111722 | | 11/17/2022 | L38535-4 |
| OW20S-1-GW-190-200-01-111722 | | 11/17/2022 | L38535-5 |
| OW20S-2-GW-200-210-01-111722 | | 11/17/2022 | L38535-6 |
| MW20B-GW-90-100-01-111722 | | 11/17/2022 | L38535-7 |
| OW20S-3-GW-165-175-01-111722 | | 11/17/2022 | L38535-8 N |

*Data validation activities were conducted with reference to:*

- *Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2022.*
- *Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.*

*Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).*

***REVIEW ELEMENTS***

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✗ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification (CCVs) |
| ✓ | Laboratory and trip blanks |
| ✗ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control samples (LCSs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes, with the exception of two results that were rejected due to severely low labeled compound recoveries. Also, results were qualified as estimated (J/UJ) with bias flags (+/-) as appropriate, due to hold times, labeled compound recoveries, and quantitation (including estimated maximum concentrations) as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

The sample was received at the laboratory intact; however, three of the samples were above the 6 °C temperature requirement at 7.8 °C. Results were not qualified as the samples were received at a temperature less than 10 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted 2 to 26 days over the 90-day hold time and detects were qualified as estimated biased low (J-), while nondetects were qualified UJ.

**Calibration**

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation. Some of the secondary transitions listed in the MPCA guidance document were not used, but the secondary transitions followed Method 1633 requirements.

Second source verification recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. The method blank results were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. Three trip blanks (TRIP-COOLER6-110722 [L38375-6]; TRIP-COOLER1-113022 [L38500-1], and TRIP-COOLER2-113022[L38500-2]) were associated with the sample data; however, they were not reported in this SDG. The trip blank results were nondetect.

**Labeled Compounds**

Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two PFBA results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZH-1-GW-90-100-01-110222 | L38383-1 | 13C4-PFBA | 22.2 | J+ |
| MW20B-GW-90-100-01-103122 | L38383-5 i | 13C4-PFBA | 4.68 | R |
| OW20P-1-GW-150-160-01-112122 | L38535-1 | 13C4-PFBA | 11.7 | J+ |
| OW20T-1-GW-98-108-01-112122 | L38535-3 | 13C4-PFBA | 49.6 | J+ |
| OW20S-2-GW-200-210-01-111722 | L38535-6 | D5-EtFOSAA | 47.0 | UJ |
| OW20S-2-GW-200-210-01-111722 | L38535-6 | 13C4-PFBA | 42.8 | J+ |
| MW20B-GW-90-100-01-111722 | L38535-7 | 13C5-PFPeA | 48.7 | J+ |
| MW20B-GW-90-100-01-111722 | L38535-7 i | 13C4-PFBA | 3.71 | R |

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was not associated with this data set.

**LCS/LCSD Results**

LCS/LCSDs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits.

## Field Duplicate Results

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

PFBA and PFOA for sample MW20B-GW-90-100-01-103122 (L38383-5); PFOA and PFOS for sample OW20T-1-GW-98-108-01-112122 (L38535-3 ); and PFBA for sample MW20B-GW-90-100-01- 111722 (L38535-7) were reported from re-analyses as the initial run indicated carryover.

Quantitations were performed to include branched isomers where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1 (primary transition); and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and are considered biased high (J+). The qualifiers for results listed below were updated to estimated (J) for samples with ion ratio exceedances (high bias) and hold time exceedances (low bias) as both high and low bias are present.

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| PZH-1-GW-90-100-01-110222 | L38383-1 | PFOS | Yes |
| OW20P-1-GW-150-160-01-112122 | L38535-1 | PFOS | Yes |
| OW20S-1-GW-190-200-01-111722 | L38535-5 | PFOS | Yes |
| OW20S-2-GW-200-210-01-111722 | L38535-6 | PFHxS | No |
| OW20S-3-GW-165-175-01-111722 | L38535-8 N | PFOS | Yes |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

Results with both high and low bias were qualified as estimated (J).

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| All Samples | All PFAS (unless listed below) | ng/L | Detects: J- Nondetects: UJ | ht |
| MW20B-GW-90-100-01-103122 (L38383-5 i)<br>MW20B-GW-90-100-01-111722 (L38535-7 i) | PFBA | ng/L | R | s |
| PZH-1-GW-90-100-01-110222 (L38383-1)<br>OW20P-1-GW-150-160-01-112122 (L38535-1)<br>OW20T-1-GW-98-108-01-112122 (L38535-3)<br>OW20S-2-GW-200-210-01-111722 (L38535-6) | PFBA | ng/L | J | s |
| OW20S-2-GW-200-210-01-111722 (L38535-6) | EtFOSAA | ng/L | UJ | s |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW20B-GW-90-100-01-111722 (L38535-7) | PFPeA | ng/L | J | s |
| PZH-1-GW-90-100-01-110222 (L38383-1)<br>OW20P-1-GW-150-160-01-112122 (L38535-1)<br>OW20S-1-GW-190-200-01-111722 (L38535-5)<br>OW20S-3-GW-165-175-01-111722 (L38535-8 N) | PFOS | ng/L | J | em |
| OW20S-2-GW-200-210-01-111722 (L38535-6) | PFHxS | ng/L | J | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)
UJ Estimated reporting limit

(2): Reason Codes:

em Estimated maximum concentration
ht Hold time
q Quantitation
s Labeled compound

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85639 (WG84134)**

**Chain of custody preservation, and holding time (Section 1C):** Ten samples were extracted 5 to 15 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. Samples results qualified are listed below:

MW14B-GW-60-70-01-102622 (L38327-65)
MW14B-GW-60-70-02-102622 (L38327-66)
MW14B-GW-60-70-03-102622 (L38327-67 (A))
BL3-WAT-BULK-01-102422 (L38375-62)
OD5-WAT-BULK-01-102422 (L38375-63)
OD12-WAT-BULK-01-102522 (L38375-64)
MW3A-GW-230-250-01-110422 (L38375-65)
MW3B-GW-110-130-01-110422 (L38375-66)
MW26C-GW-130-140-01-103122 (L38375-67)
MW27A-GW-221-222-01-102722 (L38375-68)

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detections associated with high recoveries were qualified estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. Three results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW14B-GW-60-70-01-102622 | L38327-65 | 13C2-4:2 FTS | 168 | No Qualifier |
| MW14B-GW-60-70-03-102622 | L38327-67 (A) | 13C2-4:2 FTS | 172 | No Qualifier |
| BL3-WAT-BULK-01-102422 | L38375-62 | 13C2-4:2 FTS | 154 | No Qualifier |
| OD12-WAT-BULK-01-102522 | L38375-64 | 13C4-PFBA | 37.8 | J+ |
| MW3A-GW-230-250-01-110422 | L38375-65 | 13C2-8:2 FTS | 33.5 | UJ |
| MW3A-GW-230-250-01-110422 | L38375-65 | D5-EtFOSAA | 41.2 | UJ |
| MW3A-GW-230-250-01-110422 | L38375-65 | D3-MeFOSAA | 34.7 | UJ |
| MW3A-GW-230-250-01-110422 | L38375-65 | 13C4-PFBA | 4.12 | R |
| MW3A-GW-230-250-01-110422 | L38375-65 | 13C8-PFOSA | 151 | J- |
| MW3B-GW-110-130-01-110422 | L38375-66 | 13C2-8:2 FTS | 34.8 | UJ |
| MW3B-GW-110-130-01-110422 | L38375-66 | D5-EtFOSAA | 47.2 | UJ |
| MW3B-GW-110-130-01-110422 | L38375-66 | D3-MeFOSAA | 39.9 | UJ |
| MW3B-GW-110-130-01-110422 | L38375-66 | 13C4-PFBA | 4.26 | R |
| MW26C-GW-130-140-01-103122 | L38375-67 | 13C4-PFBA | 10.4 | J+ |
| TRIP-COOLER1-113022 | L38500-1 | 13C2-4:2 FTS | 162 | No Qualifier |
| OW20J-1-GW-310-320-01-112322 | L38500-11 | D5-EtFOSAA | 49.6 | UJ |
| OW20J-1-GW-310-320-01-112322 | L38500-11 | 13C4-PFBA | 5.66 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW20J-3-GW-280-290-01-112322 | L38500-12 | 13C4-PFBA | 13.3 | J+ |
| OW20J-2-GW-310-320-01-111722 | L38500-14 | D5-EtFOSAA | 49.2 | UJ |
| OW20J-2-GW-310-320-01-111722 | L38500-14 | 13C4-PFBA | 15.3 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW14B-GW-60-70-03-102622:** | | | | | |
| PFOS | **64.2 /** 85.1 | 70-130 | 0.67 | 30 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| 11Cl-PF3OUdS | **67.0 /** 73.8 | 70-130 | 8.03 | 30 | The results for samples MW14B-GW-60-70-01-102622MW14B-GW-60-70-02-102622, and MW14B-GW-60-70-03-102622 were nondetect and qualified as estimated (UJ). |
| NFDHA | 118 / **144** | 70-130 | 18.2 | 30 | The associated results were nondetect. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW26C-GW-130-140-01-103122 | L38375-67 | PFOS |
| OW20J-3-GW-280-290-01-112322 | L38500-12 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

PFOA and PFOS results for sample MW3B-GW-110-130-01-110422 (L38375-66) were reported from 5 times dilution analyses because the concentration in the undiluted analyses exceeded the instrument calibration range.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85639 (WG84134) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/15/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Hold times are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER1-113022 (L38500-1)<br>TRIP-COOLER2-113022 (L38500-2)<br>EQ-WHALETUBE-111122 (L38500-3)<br>EQ-PERITUBE-111122 (L38500-4)<br>EQ-DIPPER-111122 (L38500-5)<br>Other trip blanks, not reported in this SDG, but associated with groundwater samples reported in this data set:<br>TRIP-COOLER7-110222<br>TRIP-COOLER7-110722 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | The trip blank results were nondetect.<br>EQ-WHALETUBE-111122 (L38500-3):<br>PFBS 5.23 ng/L<br>EQ-PERITUBE-111122 (L38500-4):<br>PFBS 3.80 ng/L<br>EQ-DIPPER-111122 (L38500-5) - no detects<br>Only field QC blanks were collected 11/11/2022. No associated samples. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | Yes | No | N/A | Comments |
|---|---|---|---|---|

| | | | | | | |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | ☒ | ☐ | ☐ | MW14B-GW-60-70-03-102622 |
| | ii. | If no, is there and explanation in the report as to why? | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | ☐ | ☒ | ☐ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MS/MSD - RPDs ok<br>Field triplicate:<br>MW14B-GW-60-70-01-102622<br>MW14B-GW-60-70-02-102622<br>MW14B-GW-60-70-03-102622 |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was use for three samples (MW14B-GW-60-70-01-102622, MW14B-GW-60-70-02-102622, and MW14B-GW-60-70-03-102622) due to high analyte concentrations.<br>In addition, the 60 mL sample bottle was consumed instead of the 500 mL sample bottle for samples BL3-WAT-BULK-01-102422 and OD5-WAT-BULK-01-102422. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | Yes | No | N/A | Comments |
|---|---|---|---|---|

| | | | | | |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Three results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/17/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85643 (WG84135)**

**Chain of custody preservation, and holding time (Section 1C):** Samples listed below were received at a temperature above the 6 °C criteria. Results were not qualified, as they were received at a temperature <10 °C.

| Sample ID | Receipt Temperature (°C) |
|---|---|
| MW2E-GW-80-90-01-102622 (L38375-16) | 8.8 |
| TRIP-COOLER5-110722 (L38375-5) | 7.2 |
| TRIP-COOLER6-110722 (L38375-6) | 7.8 |

**Chain of custody preservation, and holding time (Section 1E):** The samples were extracted 4 to 16 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+) biased high, as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. One result was rejected (R) due to a severely low labeled compound recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW18A-GW-352-362-01-110222 | L38375-9 | 13C4-PFBA | 8.0 | R |
| MW10A-GW-273-283-01-110222 | L38375-13 | 13C4-PFBA | 10.1 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside of acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW17C-GW-38.5-48.5-03-110222:** | | | | | |
| 11Cl-PF3OUdS | **61.9 / 68.4** | 70-130 | 8.18 | 30 | The results for samples MW17C-GW-38.5-48.5-01-110222, MW17C-GW-38.5-48.5-02-110222, and MW17C-GW-38.5-48.5-03-110222 were nondetect and qualified as estimated (UJ). |
| PFDoS | **62.6 /** 104 | 70-130 | 18.3 | 30 | |
| PFBA | **132 / 144** | 70-130 | 2.7 | 30 | The detects for samples MW17C-GW-38.5-48.5-01-110222, MW17C-GW-38.5-48.5-02-110222, and MW17C-GW-38.5-48.5-03-110222 were qualified as estimated biased high (J+). |
| NFDHA | 127 / **136** | 70-130 | 5.28 | 30 | The associated results were nondetect. No qualifiers. |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW18B-GW-225-235-01-110222 | L38375-8 | PFOS |
| MW18A-GW-352-362-01-110222 | L38375-9 | PFOS |
| MW10A-GW-273-283-01-110222 | L38375-13 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for sample MW2E-GW-80-90-01-102622 (L38375-16) was reported from 5 times dilution analyses because the concentration in the undiluted analyses exceeded the instrument calibration range.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85643 (WG84135) Report date (mm/dd/yyyy): 7/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/17/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Three samples were received at a temperature above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Hold times and sample receipt temperatures are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-110722 (L38375-1)<br>TRIP-COOLER2-110722 (L38375-2)<br>TRIP-COOLER3-110722 (L38375-3)<br>TRIP-COOLER4-110722 (L38375-4)<br>TRIP-COOLER5-110722 (L38375-5)<br>TRIP-COOLER6-110722 (L38375-6)<br>TRIP-COOLER7-110722 (L38375-7) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW17C-GW-38.5-48.5-03-110222 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MS/MSD - RPDs ok<br>Field triplicate:<br>MW17C-GW-38.5-48.5-01-110222 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | MW17C-GW-38.5-48.5-02-110222<br>MW17C-GW-38.5-48.5-03-110222 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One result was rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/17/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85866 (WG84135)**

**Chain of custody preservation, and holding time (Section 1E):** The samples were extracted 1 to 5 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ). Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZED-GW-16-21-01-012323 | L38792-61 | D3-MeFOSAA | 46.1 | UJ |
| PZED-GW-16-21-01-012323 | L38792-61 | D5-EtFOSAA | 49.8 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85031-103 (Low Level) | N-EtFOSAA | 65.5 | 70-130 | Associated detects were qualified as estimated biased low (J-), and nondetect results were qualified UJ. |
| | N-MeFOSA | 57.1 | 70-130 | |
| | N-EtFOSA | 56.6 | 70-130 | |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside of acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW14B-GW-60-70-03-012723:** | | | | | |
| PFOA | **59.2** / 80.0 | 70-130 | 1.86 | 30 | The associated sample concentrations were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **NC** / 72.4 | 70-130 | 1.99 | 30 | |

NC – Not calculated
**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZGS-GW-20-30-01-012423 | L38792-65 | PFOS |
| PZGS-GW-20-30-02-012423 | L38792-66 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for samples PZED-GW-16-21-01-012323 (L38792-61), PZES-GW-6-11-01-012323 (L38792-62), PZFS-GW-18-28-01-012423 (L38792-64), and PZHD-GW-21-26-01-012423 (L38792-67)

were reported from 5 times dilution analyses because the concentration in the undiluted analyses exceeded the instrument calibration range.

Detects with both low bias and qualifications due to estimated maximum concentration were qualified as estimated (J).

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# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85866 (WG85031) Report date (mm/dd/yyyy): 6/9/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/23/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperatures are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER3-013023<br>TRIP-COOLER5-013023<br>TRIP-COOLER6-013023<br>TRIP-COOLER7-013023 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW14B-GW-60-70-03-012723 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifiers. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MS/MSD - RPDs ok<br>Field triplicate:<br>MW14B-GW-60-70-01-012723<br>MW14B-GW-60-70-02-012723<br>MW14B-GW-60-70-03-012723 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | Field duplicate:<br>PL41-GW-72-82-01-012323<br>PL41-GW-72-82-02-012323<br>Field duplicate:<br>PZGS-GW-20-30-01-012423<br>PZGS-GW-20-30-02-012423 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was use for three samples (MW14B-GW-60-70-01-012723, MW14B-GW-60-70-02-012723, and MW14B-GW-60-70-03-012723 ) due to high analyte concentrations. In addition, a reduced sample volume was used for sample SP42-GW-116-126-01-012323 due to high suspended solids. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/27/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85891 (WG85032)**

**Chain of custody preservation, and holding time (Section 1C):** Ten samples were extracted 1 to 3 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. Samples results qualified are listed below:

| Sample ID | Lab ID |
|---|---|
| PZHS-GW-6-16-01-012423 | L38792-68 |
| PZID-GW-22-27-01-012323 | L38792-69 |
| OD4-WAT-BULK-01-012523 | L38792-70 |
| OD5-WAT-BULK-01-012523 | L38792-71 |
| WL6-WAT-BULK-01-012523 | L38792-72 |
| WL11-WAT-BULK-01-012523 | L38792-73 |
| BL5-WAT-BULK-01-012523 | L38792-74 |
| BL5-WAT-BULK-02-012523 | L38792-75 |
| RC5-WAT-BULK-01-012523 | L38792-76 |
| RC5-WAT-BULK-02-012523 | L38792-77 |
| RC5-WAT-BULK-03-012523 | L38792-78 (A) |
| EP16-WAT-BULK-01-012523 | L38792-79 |
| EP19-WAT-BULK-01-012523 | L38792-80 |

**Labeled Analogs - surrogates (Section 4Bii** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD4-WAT-BULK-01-012523 | L38792-70 | d9-NEt-FOSE | 37.0 | UJ |
| OD4-WAT-BULK-01-012523 | L38792-70 | 13C4-PFBA | 31.2 | J+ |
| OD5-WAT-BULK-01-012523 | L38792-71 | D5-EtFOSAA | 39.2 | UJ |
| WL6-WAT-BULK-01-012523 | L38792-72 | D5-EtFOSAA | 47.1 | UJ |
| WL6-WAT-BULK-01-012523 | L38792-72 | D5-N-EtFOSA | 47.5 | UJ |
| WL6-WAT-BULK-01-012523 | L38792-72 | 13C4-PFBA | 25.2 | J+ |
| WL6-WAT-BULK-01-012523 | L38792-72 | 13C2-PFDoA | 33.0 | UJ |
| WL6-WAT-BULK-01-012523 | L38792-72 | 13C8-PFOS | 39.8 | J+ |
| WL11-WAT-BULK-01-012523 | L38792-73 | 13C4-PFBA | 30.5 | J+ |
| BL5-WAT-BULK-01-012523 | L38792-74 | 13C4-PFBA | 17.8 | J+ |
| BL5-WAT-BULK-02-012523 | L38792-75 | d9-NEt-FOSE | 49.9 | UJ |
| BL5-WAT-BULK-02-012523 | L38792-75 | 13C4-PFBA | 12.5 | J+ |
| RC5-WAT-BULK-01-012523 | L38792-76 | d9-NEt-FOSE | 42.8 | UJ |
| RC5-WAT-BULK-03-012523 | L38792-78 (A) | d9-NEt-FOSE | 26.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP16-WAT-BULK-01-012523 | L38792-79 | 13C4-PFBA | 40.0 | J+ |
| EP19-WAT-BULK-01-012523 | L38792-80 | 13C4-PFBA | 26.0 | J+ |

**MS/MSD Exceedances (Section 6Av and B):** MS/MSD recoveries and relative percent differences (RPDs) were outside of acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-012523:** | | | | | |
| PFBA | **223 / 568** | 70-130 | **35.3** | 30 | The associated sample concentrations were greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **194 / 233** | 70-130 | 1.29 | 30 | |
| PFOS | **NC /**119 | 70-130 | 1.44 | 30 | |
| PFPeA | 117 **/ 146** | 70-130 | 14.9 | 30 | The results for samples RC5-WAT-BULK-01-012523, RC5-WAT-BULK-02-012523, and RC5-WAT-BULK-03-012523 were qualified as estimated biased high (J+). |
| PFHxA | **192 / 219** | 70-130 | 5.02 | 30 | |
| PFHpA | 129 **/ 135** | 70-130 | 2.63 | 30 | |
| PFBS | **131 /161** | 70-130 | 12.8 | 30 | |
| PFDoS | **18.7** / 81.2 | 70-130 | **126** | 30 | The results for samples RC5-WAT-BULK-01-012523, RC5-WAT-BULK-02-012523, and RC5-WAT-BULK-03-012523 were nondetect and qualified UJ. |
| 11Cl-PF3OUdS | **45.9 /** 80.4 | 70-130 | **56.3** | 30 | |

NC – Not calculated
**Bold** indicates an exceedance

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **RC5-WAT-BULK-01-012523 / RC5-WAT-BULK-02-012523 / RC5-WAT-BULK-03-012523:** | | | | |
| PFBA | 363 | 252 | 227 | 48.5 |
| PFPeA | 22.1 | 17 | 15.2 | 38.1 |
| **BL5-WAT-BULK-01-012523 / BL5-WAT-BULK-02-012523:** | | | | |
| PFBA | 208 | 145 | -- | 35.7 |

**10. Additional comments about lab report:**

Sample PZID-GW-22-27-01-012323 (L38792-69) was re-analyzed to confirm possible instrumental carry-over for PFHxS. The re-analysis data showed that the original analysis data was affected by carry over and the re-analysis result for PFHxS was reported.

Results PFTrDA, PFTeDA, N-MeFOSE, and N-EtFOSE for sample WL6-WAT-BULK-01-012523 (L38792-72) were reported as not quantifiable (NQ) in the laboratory report. These results were qualified as rejected (R).

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for samples PZHS-GW-6-16-01-012423 (L38792-68), RC5-WAT-BULK-01-012523 (L38792-76), RC5-WAT-BULK-02-012523 (L38792-77), RC5-WAT-BULK-03-012523 (L38792-78), and MW2F-GW-130-150-01-020223 (L38860-4) were reported from dilution analyses because the concentration in the undiluted analyses exceeded the instrument calibration range.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85891 (WG85032) Report date (mm/dd/yyyy): 6/13/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/28/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperatures (acceptable) are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-021423<br>Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER7-013023<br>TRIP-COOLER8-013023<br>Equipment blanks:<br>EQ-WHALE-TUBE-020223<br>EQ-PERI-TUBE-020223 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | The trip blank results were nondetect.<br>EQ-WHALE-TUBE-020223<br>PFOS 0.977 J ng/L<br>EQ-PERI-TUBE-020223 - no detects |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | The associated PFOS sample concentration was greater than 10 times the blank concentration. No qualifiers. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-012523 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MS/MSD - RPDs high for three compounds.<br>Field triplicate:<br>RC5-WAT-BULK-01-012523<br>RC5-WAT-BULK-02-012523<br>RC5-WAT-BULK-03-012523<br>FIeld Duplicate:<br>BL5-WAT-BULK-01-012523<br>BL5-WAT-BULK-02-012523 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± QL, used for water matrix.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Four results for sample WL6-WAT-BULK-01-012523 (L38792-72) were reported as not quantifiable (NQ), and were rejected (R).**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/28/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85913 (WG85720)**

**Chain of custody preservation, and holding time (Section 1C):** The sample was extracted 43 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZH-1-GW-90-100-01-01272 | L38792-57 R2 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both low bias and qualifications due to estimated maximum concentration were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85913 (WG85720) Report date (mm/dd/yyyy): 6/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/28/2023

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☒ | ☐ | ☐ | Sample receipt temperatures (acceptable) and hold times exceedances are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | The associated trip blank was not reported in this SDG:<br>TRIP-COOLER6-013023 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | No MS/MSDs for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD - RPDs ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Lower sample volume used due to high solids. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/28/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85917 (WG85413)**

**Chain of custody preservation, and holding time (Section 1C):** All samples except TRIP-COOLER2-042623 (L39228-2) were received at temperatures above the < 6 °C criteria, and ranged from 6.1°C to 9.3 °C. Results were not qualified, as they were received at a temperature < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZED-GW-16-21-01-032923 | L39113-27 | D3-MeFOSAA | 49.4 | UJ |
| TRIP-COOLER5-042623 | L39228-5 | D3-N-MeFOSA | 46.8 | UJ |
| TRIP-COOLER5-042623 | L39228-5 | D5-N-EtFOSA | 48.4 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **PZED-GW-16-21-03-032923:** | | | | | |
| PFOS | **542 / 284** | 70-130 | 3.79 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOA | 129 / **62.9** | 70-130 | 2.14 | 30 | |
| 11Cl-PF3OUdS | **68.3 /** 80.1 | 70-130 | 16.6 | 30 | The results for samples PZED-GW-16-21-01-032923, PZED-GW-16-21-02-032923, and PZED-GW-16-21-03-032923 were nondetect and qualified as estimated (UJ). |

**Bold indicates an exceedance**

**10. Additional comments about lab report:**

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS results for six samples were reported from 5 times dilution analyses due to high sample concentrations.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85917 (WG85413) Report date (mm/dd/yyyy): 6/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 8/29/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples except TRIP-COOLER2-042623 (L39228-2) were received at temperatures above the < 6 °C criteria, and ranged from 6.1°C to 9.3 °C. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-042623<br>TRIP-COOLER2-042623<br>TRIP-COOLER3-042623<br>TRIP-COOLER4-042623<br>TRIP-COOLER5-042623<br>TRIP-COOLER6-042623<br>TRIP-COOLER7-042623<br>TRIP-COOLER8-042623<br>Also, trip blanks associated with groundwater samples were not reported in this SDG:<br>TRIP-COOLER2-040523 (L39113-2)<br>TRIP-COOLER3-040523 (L39113-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results were nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZED-GW-16-21-03-032923 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD - RPDs ok<br>Field triplicate:<br>PZED-GW-16-21-01-032923<br>PZED-GW-16-21-02-032923<br>PZED-GW-16-21-03-032923 |
| | i. | | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | A reduced sample volume was use for sample MW14B-GW-60-70-01-032923 (L39113-23) due to high analyte concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 8/29/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85920 (WG85412)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at temperatures of 6.2 °C and 6.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC12-WAT-BULK-01-032923 | L39113-14 | 13C4-PFBA | 42.0 | J+ |
| RC12-WAT-BULK-01-032923 | L39113-14 | 13C2-PFTeDA | 49.0 | UJ<br>PFTrDA: UJ |
| EP26D-WAT-BULK-01-032923 | L39113-17 | 13C2-PFTeDA | 47.0 | UJ<br>PFTrDA: UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-033023:** | | | | | |
| PFOS | **347 / 203** | 70-130 | 4.55 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:**

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for sample RC20-WAT-BULK-01-032923 (L39113-15) was reported from a 5 times dilution analysis due to a high sample concentration.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85920 (WG85412) Report date (mm/dd/yyyy): 9/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/11/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at temperatures of 6.2 °C and 6.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-040523 (L39113-1)<br>TRIP-COOLER2-040523 (L39113-2)<br>TRIP-COOLER3-040523 (L39113-3)<br>EQ-DIPPER-032923 (L39113-4)<br>FIELD-BLANK-032923 (L39113-5)<br>EQ-PERI-032923 (L39113-6)<br>EQ-WHALE-032923 (L39113-7)) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip and equuipment blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-033023 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications required. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field triplicate:<br>RC5-WAT-BULK-01-033023 (L39113-11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | RC5-WAT-BULK-02-033023 (L39113-12)<br>RC5-WAT-BULK-03-033023 (L39113-13 (A)) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volume was use for samples RC3-WAT-BULK-01-033023 (L39113-10), RC5-WAT-BULK-01-033023 (L39113-11), RC5-WAT-BULK-02-033023 (L39113-12), and RC5-WAT-BULK-03-033023 (L39113-13 (A)) due to high analyte concentrations.<br>A reduced sample volume was use for sample EP24-WAT-BULK-01-032923 (L39113-16) due to high particulates that would have clogged to SPE column. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/11/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85935 (WG85028)**

**Chain of custody preservation, and holding time (Section 1C):** The samples were extracted 2 to 8 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85028-102 | NFDHA | 133 | 70-130 | Associated results were nondetect, and did not require qualification. |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW2D-GW-7-17-03-012423:** | | | | | |
| PFOS | **Not reported** | 70-130 | 25.5 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW3A-GW-230-250-01-012623 | L38792-13 | PFOS |
| OW7S-1-GW-90-100-01-012723 | L38792-18 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85935 (WG85028) Report date (mm/dd/yyyy): 6/15/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/13/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-013023 (L38792-1)<br>TRIP-COOLER2-013023 (L38792-2)<br>TRIP-COOLER3-013023 (L38792-3)<br>TRIP-COOLER4-013023 (L38792-4)<br>TRIP-COOLER5-013023 (L38792-5)<br>TRIP-COOLER6-013023 (L38792-6)<br>TRIP-COOLER7-013023 (L38792-7)<br>TRIP-COOLER8-013023 (L38792-8)<br>EQ-DIPPER-012523 (L38792-9) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip and equuipment blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2D-GW-7-17-03-012423 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications required. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field triplicate:<br>MW2D-GW-7-17-01-012423 (L38792-10)<br>MW2D-GW-7-17-02-012423 (L38792-11)<br>MW2D-GW-7-17-03-012423 (L38792-12 (A)) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was use for samples MW3A-GW-230-250-01-012623 (L38792-13), MW3B-GW-110-130-01-012623 (L38792-14), OW7S-1-GW-90-100-01-012723 (L38792-18), and MW8A-GW-202-212-01-012723 (L38792-20) due to high particulates.that would have clogged to SPE column. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/13/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85936 (WG85418)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at temperatures of 7.3 °C, 7.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One PFBA result was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PL41-GW-72-82-01-041723 | L39228-92 | 13C2-PFTeDA | 35.3 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-01-041723 | L39228-92 | D5-N-EtFOSA | 44.7 | UJ |
| PL41-GW-72-82-01-041723 | L39228-92 | D5-EtFOSAA | 48.6 | UJ |
| PL41-GW-72-82-01-041723 | L39228-92 | d7-NMe-FOSE | 44.5 | UJ |
| PL41-GW-72-82-01-041723 | L39228-92 | d9-NEt-FOSE | 34.9 | UJ |
| PL41-GW-72-82-02-041723 | L39228-93 | 13C4-PFBA | 40.6 | J+ |
| SP42-GW-116-126-01-041723 | L39228-94 | 13C4-PFBA | 8.55 | R |
| SP42-GW-116-126-01-041723 | L39228-94 | 13C2-PFTeDA | 15.7 | UJ<br>PFTrDA: J+ |
| SP42-GW-116-126-01-041723 | L39228-94 | 13C2-8:2 FTS | 28.4 | UJ |
| SP42-GW-116-126-01-041723 | L39228-94 | D3-N-MeFOSA | 42.0 | UJ |
| SP42-GW-116-126-01-041723 | L39228-94 | D5-N-EtFOSA | 28.2 | UJ |
| SP42-GW-116-126-01-041723 | L39228-94 | D3-MeFOSAA | 30.1 | J+ |
| SP42-GW-116-126-01-041723 | L39228-94 | D5-EtFOSAA | 33.6 | J+ |
| SP42-GW-116-126-01-041723 | L39228-94 | d7-NMe-FOSE | 45.6 | UJ |
| SP42-GW-116-126-01-041723 | L39228-94 | d9-NEt-FOSE | 38.9 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85418-103 (Low Level) | 6:2 FTS | 218 | 70-130 | Associated low level detections were qualified as estimated biased high (J+):<br><br>SP42-GW-116-126-01-041723 (L39228-94) |
| | PFHxA | 139 | 70-130 | Associated low level detections were qualified as estimated biased high (J+):<br><br>SP42-GW-116-126-01-041723 (L39228-94) |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-042423:** | | | | | |
| PFOA | **Not reported / 151** | 70-130 | 6.49 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **Not reported** | 70-130 | 1.09 | 30 | |

**Bold** indicates an exceedance

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **PL41-GW-72-82-01-041723 / PL41-GW-72-82-02-041723:** | | | |
| PFPeS | 5.83 | 0.376 U | > ± 2x LOQ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZGD-GW-35-40-01-041923 | L39228-86 | PFOS |
| SP42-GW-116-126-01-041723 | L39228-94 | PFTrDA |
| SP42-GW-116-126-01-041723 | L39228-94 | PFDoA |
| W6201-GW-109-204-01-042423 | L39228-95 | PFTrDA |
| W6201-GW-109-204-01-042423 | L39228-95 | PFNS |
| W6201-GW-109-204-01-042423 | L39228-95 | PFDA |
| W6201-GW-109-204-01-042423 | L39228-95 | PFDoA |
| W6102-GW-93-105-01-041923 | L39228-96 | PFNA |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Eight results were reported from five times dilution analyses due to high concentrations.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85936 (WG85418) Report date (mm/dd/yyyy): 6/16/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/14/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at temperatures of 7.3 °C, 7.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER7-042623 (L39228-7)<br>TRIP-COOLER8-042623 (L39228-8)<br>TRIP-COOLER9-042623 (L39228-9) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-042423 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications required. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field Duplicate:<br>PL41-GW-72-82-01-041723<br>PL41-GW-72-82-02-041723<br>Field triplicate: |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | RC5-WAT-BULK-01-042423<br>RC5-WAT-BULK-02-042423<br>RC5-WAT-BULK-03-042423 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± QL, used for water matrix.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was use for samples RC3-WAT-BULK-01-042423 (L39228-97), RC5-WAT-BULK-01-041923 (L39228-98), RC5-WAT-BULK-01-042423 (L39228-99), RC5-WAT-BULK-02-042423 (L39229-1), RC5-WAT-BULK-03-042423 (L39229-2(A)), and RC22-WAT-BULK-01-042423 (L39229-3) due to high analyte concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One PFBA result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/15/2023

**SGS AXYS DPWG85618** 11/27/23 **LS**

Initial Cal 2/19/23 **PFOS**

| Area Counts | | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form |
|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS SUM | | | |
| | | | | |
| 26900 | 1525 | 0.4 | 1.4261 | 1.43 |
| 37502 | 3424 | 0.8 | 1.1483 | 1.15 |
| 24441 | 5305 | 2 | 1.0920 | 1.09 |
| 27848 | 14531 | 5 | 1.0501 | 1.05 |
| 31415 | 35990 | 10 | 1.1527 | 1.15 |
| 35425 | 86522 | 20 | 1.2288 | 1.23 |
| 33752 | 183693 | 50 | 1.0952 | 1.10 |
| 26633 | 750554 | 250 | 1.1342 | 1.13 |
| | | Avg: | 1.1659 | 1.17 |
| | | %RSD: | 10.0963 | 10.20 |

p 306

1.16597 (raw data)

Internal Std Conc: 10.062

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| R1-GW-75-90-01-033023 | PFOS | 2414 | 25903 | 1 | 199.56 | 199 | 0.0040 | 0.004 | L39114-1 |
| W33-GW-12-15.5-01-033023 | PFOS | 224066 | 25131 | 1 | 3885957 | 3890000 | 0.0000198 | 0.004 | L39114-2 |
| WG85719-102 | PFOS | 14746 | 25758 | 1 | 4.94 | 4.94 | 1.00 | 0.004 | |
| WG85719-104 | PFOS | 14418 | 26270 | 1 | 4.74 | 4.74 | 1.00 | 0.004 | |
| WG85719-103 | PFOS | 4959 | 25850 | 1 | 1.66 | 1.66 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 6/7/23 15:59 | 21187 | 39964 | 4.575 | 4.55 | 5.00 | 91.00 | 91.1% |
| CCV 6/7/23 18:52 | 21582 | 40761 | 4.569 | 4.57 | 5.00 | 91.40 | 91.4% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank (WG85719-101) | PFOS | 0.016 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG85938 (Analysis WG85719) dated 6/20/2023 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 12/5/2023 |

The groundwater samples listed below were collected at the Project 1007 Site March 30, 2023.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| R1-GW-75-90-01-033023 | | 3/30/2023 | L39114-1 |
| W33-GW-12-15.5-01-033023 | | 3/30/2023 | L39114-2 |

*Data validation activities were conducted with reference to:*

- *Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2023.*
- *Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.*

*Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).*

***REVIEW ELEMENTS***

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✓ | Initial calibration/continuing calibration verification (CCVs) |
| ✓ | Laboratory and trip blanks |
| ✓ | Labeled Compounds |
| NA | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✓ | Laboratory control samples/Laboratory Control Sample Duplicates (LCS/LCSDs) |
| NA | Field duplicates |
| ✗ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

## SUMMARY

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Results were qualified as estimated (J) with bias flags (+/-) as appropriate, due to ion ratio as shown in Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

## DETAILED REVIEW

### Data Completeness

The analytical data package was verified as complete.

### Sample Receipt and Holding Times

The samples were received at the laboratory intact; however, were above the 6 °C temperature requirement at 6.5 °C. Results were not qualified as the samples were received at a temperature less than 10 °C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. Samples were extracted within the 90-day hold time.

### Calibration

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation. Some of the secondary transitions listed in the MPCA guidance document were not used, but the secondary transitions followed Method 1633 requirements.

Second source verification recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

### Laboratory and Trip Blanks

Method blanks were analyzed to assess laboratory contamination. 6:2 FTS was detected in the method blank at a concentration of 24100 ng/l. 6:2 FTS was not detected in the samples. The method blank concentration was calculated using sample size similar to the samples. The majority of the time this is 500 mls (the standard sample size); however, in this case 1 ml was used. The absolute (on-column) concentration was 24.07 ng. The laboratory indicated this detect was an isolated occurrence. Historical method blank data was provided for blank runs from 05/01/2023 through to 07/31/2023, and this data showed that the majority of the method blank results for 6:2 FTS were nondetect.

Trip blanks are used to assess contamination that occurs during sample shipping. One trip blank (TRIP-COOLER2-040523 [L39113-2] was associated with the sample data; however, was not reported in this SDG. The trip blank results were nondetect.

**Labeled Compounds**

Labeled compound recoveries were within MPCAs recovery limits of 100 ± 50%.

**MS/MSD Results**

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. An MS/MSD was not associated with this data set.

**LCS/LCSD Results**

LCS/LCSDs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS/LCSD %Rs and relative percent differences (RPDs) were within the MPCA limits, with the exceptions listed in the table below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85719-103 (Low Level) | 6:2 FTS | 177 | 70-130 | Associated results were nondetect, and were acceptable without qualification. |

LCS results were recalculated for PFOS during validation. Results reported are verified to be correct.

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Field duplicates were not associated with this data set.

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Quantitations were performed to include branched isomers where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1 (primary transition); and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet ion ratio criteria and are considered biased high (J+).

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| R1-GW-75-90-01-033023 | L39114-1 | PFHxS | No |
| W33-GW-12-15.5-01-033023 | L39114-2 | PFHxS | No |
| W33-GW-12-15.5-01-033023 | L39114-2 | PFOS | No |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

**Qualified Analytical Results**

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| R1-GW-75-90-01-033023 (L39114-1)<br>W33-GW-12-15.5-01-033023 (L39114-2) | PFHxS | ng/L | J+ | q |
| W33-GW-12-15.5-01-033023 (L39114-2) | PFOS | ng/L | J+ | q |

(1): Data Validation Qualifiers:

J Estimated (+/- indicates high or low bias)

(2): Reason Codes:

q Quantitation (ion ratios)

# Attachment A

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85965 (WG85417)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at temperatures of 6.1 °C, 8.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One PFBA result was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW20J-1-GW-310-320-01-041723 | L39228-68 | d9-NEt-FOSE | 49.8 | UJ |
| OW20J-1-GW-310-320-01-041723 | L39228-68 | d7-NMe-FOSE | 47.3 | UJ |
| OW20J-1-GW-310-320-01-041723 | L39228-68 | D3-N-MeFOSA | 39.3 | UJ |
| OW20J-1-GW-310-320-01-041723 | L39228-68 | D5-N-EtFOSA | 37.9 | UJ |
| OW20J-3-GW-280-290-01-041723 | L39228-69 | 13C4-PFBA | 14.6 | J+ |
| OW20P-1-GW-150-160-01-041723 | L39228-70 | 13C4-PFBA | 4.43 | R |
| PZH-1-GW-90-100-01-041923 | L39228-72 | 13C2-PFDoA | 48.8 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | 13C2-PFTeDA | 38.7 | UJ<br>PFTrDA: UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | D3-N-MeFOSA | 47.0 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | D5-N-EtFOSA | 44.9 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | D3-MeFOSAA | 46.6 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | D5-EtFOSAA | 44.2 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | d7-NMe-FOSE | 48.4 | UJ |
| PZH-1-GW-90-100-01-041923 | L39228-72 | d9-NEt-FOSE | 48.9 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW20J-3-GW-280-290-01-041723 | L39228-69 | PFOS |
| OW20P-1-GW-150-160-01-041723 | L39228-70 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85965 (WG85417) Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/17/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at temperatures of 6.1 °C, 8.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER5-042623 (L39228-5)<br>TRIP-COOLER6-042623 (L39228-6)<br>TRIP-COOLER7-042623 (L39228-7) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD reported |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD - RPDs ok<br>Field Duplicate:<br>PZED-GW-16-21-01-041723<br>PZED-GW-16-21-02-041723 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for 13 samples due to high suspended solids. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One PFBA result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85967 (WG85414)**

**Chain of custody preservation, and holding time (Section 1C):** All samples, except for three, were received at temperatures of 7.3 °C, 7.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One PFBA result was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2B-GW-57-62-01-042123 | L39228-16 | 13C4-PFBA | 38.8 | J+ |
| MW2C-GW-35-40-01-042123 | L39228-19 | 13C4-PFBA | 32.1 | J+ |
| MW4A-GW-140-160-01-041923 | L39228-24 | 13C4-PFBA | 17.4 | J+ |
| MW5B-GW-110-120-01-041923 | L39228-26 | 13C4-PFBA | 5.26 | R |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW2B-GW-57-62-03-042123:** | | | | | |
| PFOA | **Not reported / 163** | 70-130 | 7.03 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **179 / 469** | 70-130 | 4.52 | 30 | |
| 3:3 FTCA | 107 **/ 58.8** | 70-130 | **61.9** | 30 | Detects for samples MW2B-GW-57-62-01-042123, MW2B-GW-57-62-02-042123, and MW2B-GW-57-62-03-042123 were qualified as estimated biased low (J-), and nondetect were qualified UJ. |
| PFMPA | 102 **/ 53.5** | 70-130 | **54.1** | 30 | |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW3A-GW-230-250-01-042123 | L39228-22 | PFDS |
| MW3B-GW-110-130-01-042123 | L39228-23 | PFNS |
| MW5A-GW-210-220-01-042423 | L39228-25 | PFTrDA |
| MW5A-GW-210-220-01-042423 | L39228-25 | PFOS |
| MW5B-GW-110-120-01-041923 | L39228-26 | PFNS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85967 (WG85414) Report date (mm/dd/yyyy): 6/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/18/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | All samples, except for three, were received at temperatures of 7.3 °C, 7.6 °C, and 8.9 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | TRIP-COOLER9-042623 (L39228-9)<br>TRIP-COOLER10-042623 (L39228-10)<br>TRIP-COOLER11-042623 (L39228-11)<br>EQ-WHALETUBE-01-042123 (L39228-12)<br>EQ-PERITUBE-01-042123 (L39228-13)<br>EQ-DIPPER-01-042123 (L39228-14)<br>Trip blanks associated with groundwater samples in this SDG (not reported in this SDG):<br>TRIP-COOLER1-042623 (L39228-1)<br>TRIP-COOLER2-042623 (L39228-2) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The equipment and trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | One detect (below quantitation limit):<br>6:2 FTS 1.47 J ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2B-GW-57-62-03-042123 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: See data qualifications summary (attachment) for exceedances.<br>Field Triplicate:<br>MW2B-GW-57-62-01-042123<br>MW2B-GW-57-62-02-042123<br>MW2B-GW-57-62-03-042123 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for sample MW3A-GW-230-250-01-042123 (L39228-22) due to high suspended solids. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One PFBA result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85968 (WG85415)**

**Chain of custody preservation, and holding time (Section 1C):** Ten samples were received at a temperature of 9.3 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Three PFBA results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW9B-GW-90-100-01-041823 | L39228-33 | 13C4-PFBA | 5.44 | R |
| MW10B-GW-130-140-01-041923 | L39228-35 | 13C4-PFBA | 4.30 | R |
| MW13B-GW-295-305-01-042123 | L39228-38 | 13C4-PFBA | 7.70 | R |
| MW14A-GW-316-326-01-041823 | L39228-41 | 13C4-PFBA | 42.5 | J+ |
| MW17B-GW-90-100-01-041923 | L39228-44 | D5-N-EtFOSA | 47.1 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW6C-GW-35-40-03-041723:** | | | | | |
| PFBA | **219 / 261** | 70-130 | 1.78 | 30 | The sample concentrations were greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **142 / 165** | 70-130 | 2.24 | 30 | |
| PFOS | **217 / 254** | 70-130 | 1.76 | 30 | |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW6C-GW-35-40-02-041723 | L39228-28 | PFDA |
| MW10B-GW-130-140-01-041923 | L39228-35 | PFDoA |
| MW10C-GW-10-20-01-041923 | L39228-36 | PFOS |
| MW13B-GW-295-305-01-042123 | L39228-38 | PFOS |
| MW13C-GW-115-125-01-042123 | L39228-39 | PFOS |
| MW13D-GW-15-25-01-042123 | L39228-40 | PFOS |
| MW17B-GW-90-100-01-041923 | L39228-44 | PFOS |

The PFBA result for sample MW17B-GW-90-100-01-041923 (L39228-44 N) was reported from five times dilution analyses due to high concentrations.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG85968 (WG85415) Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/28/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Ten samples were received at a temperature of 9.3 °C, and above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER2-042623 (L39228-2)<br>TRIP-COOLER3-042623 (L39228-3) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW6C-GW-35-40-03-041723 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: RPDs ok<br>Field Duplicate:<br>MW10C-GW-10-20-01-041923<br>MW10C-GW-10-20-02-041923<br>Field Triplicate:<br>MW6C-GW-35-40-01-041723<br>MW6C-GW-35-40-02-041723 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | MW6C-GW-35-40-03-041723 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for samples MW8A-GW-202-212-01-041823 (L39228-31) and MW10A-GW-273-283-01-041923 (L39228-34) due to high solids. A reduced sample volume was used for samples MW14B-GW-60-70-01-041823 (L39228-42) and MW14D-GW-6-21-01-041823 (L39228-43) due to high analyte concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Three PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/18/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85973 (WG85416)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at temperatures of 6.1 °C, 7.6 °C, and 9.3 °C, and were above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two PFBA results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW18A-GW-352-362-01-041823 | L39228-48 | 13C4-PFBA | 16.3 | J+ |
| MW20A-GW-130-140-01-041823 | L39228-50 | d9-NEt-FOSE | 45.0 | UJ |
| MW20B-GW-90-100-01-041723 | L39228-51 | 13C4-PFBA | 3.48 | R |
| MW20B-GW-90-100-01-041723 | L39228-51 | 13C5-PFPeA | 48.0 | J+ |
| MW22B-GW-290-300-01-041823 | L39228-54 | 13C2-8:2 FTS | 49.0 | UJ |
| MW22B-GW-290-300-01-041823 | L39228-54 | 13C4-PFBA | 15.1 | J+ |
| MW26A-GW-380-390-01-041923 | L39228-56 | 13C4-PFBA | 44.1 | J+ |
| MW27B-GW-120-130-01-041923 | L39228-60 | 13C4-PFBA | 40.3 | J+ |
| OW5O-1-GW-200-210-01-041923 | L39228-61 | 13C4-PFBA | 6.42 | R |
| OW7Q-1-GW-64-74-01-041823 | L39228-62 | d9-NEt-FOSE | 14.3 | UJ |
| OW7Q-1-GW-64-74-01-041823 | L39228-62 | d7-NMe-FOSE | 34.7 | UJ |
| OW7S-1-GW-90-100-01-041823 | L39228-63 | 13C4-PFBA | 43.3 | J+ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW17C-GW-38.5-48.5-03-041923 | L39228-47 (A) | PFPeS |
| MW18A-GW-352-362-01-041823 | L39228-48 | PFOS |
| MW18B-GW-225-235-01-041823 | L39228-49 | PFOS |
| MW26C-GW-130-140-01-041923 | L39228-58 | PFOS |
| MW27B-GW-120-130-01-041923 | L39228-60 | PFOS |
| OW7S-1-GW-90-100-01-041823 | L39228-63 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Sample MW26B-GW-249-259-01-042123 (L39228-57) was re-analyzed for PFHxA and PFOA due to carryover.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG85973 (WG85416) Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 9/29/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at temperatures of 6.1 °C, 7.6 °C, and 9.3 °C, and were above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER3-042623 (L39228-3)<br>TRIP-COOLER4-042623 (L39228-4)<br>TRIP-COOLER5-042623 (L39228-5) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW17C-GW-38.5-48.5-03-041923 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: RPDs ok<br>Field Triplicate:<br>MW17C-GW-38.5-48.5-01-041923<br>MW17C-GW-38.5-48.5-02-041923<br>MW17C-GW-38.5-48.5-03-041923 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Two PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 9/29/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG85975 (WG85419)**

**Chain of custody preservation, and holding time (Section 1C):** Four samples were received at a temperature of 7.6 °C, and were above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One PFBA result was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26D-WAT-BULK-01-041923 | L39229-9 | 13C4-PFBA | 20.4 | J+ |
| EP26D-WAT-BULK-01-042423 | L39229-10 | 13C4-PFBA | 12.2 | J+ |
| EP21-WAT-BULK-01-042423 | L39229-11 | 13C4-PFBA | 32.6 | J+ |
| EP16-WAT-BULK-01-042423 | L39229-12 | 13C4-PFBA | 6.78 | R |
| WL6-WAT-BULK-01-041923 | L39229-13 | 13C4-PFBA | 34.1 | J+ |
| WL6-WAT-BULK-01-042423 | L39229-14 | 13C4-PFBA | 13.5 | J+ |
| WL15-WAT-BULK-01-042423 | L39229-15 | 13C4-PFBA | 36.1 | J+ |
| OD3-WAT-BULK-01-042423 | L39229-16 | 13C4-PFBA | 42.5 | J+ |
| OD5-WAT-BULK-01-042423 | L39229-18 | 13C4-PFBA | 17.9 | J+ |
| BL5-WAT-BULK-01-042423 | L39229-20 | 13C4-PFBA | 33.7 | J+ |
| BL5-WAT-BULK-02-042423 | L39229-21 | 13C4-PFBA | 23.7 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85419-103 (Low Level) | NFDHA | **143** | 70-130 | Associated results were nondetect, and were acceptable without qualification. |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| EP16-WAT-BULK-01-042423 | L39229-12 | N-EtFOSAA |
| OD4-WAT-BULK-01-042423 | L39229-17 | PFOS |
| BL3-WAT-BULK-01-042423 | L39229-19 | PFNA |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

PFOS results for four samples were reported from a five times dilution analysis due to high analyte concentrations.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG85975 (WG85419) Report date (mm/dd/yyyy): 6/20/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/2/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Four samples were received at a temperature of 7.6 °C, and were above the < 6 °C criteria. Results were not qualified, as they were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER9-042623 (L39228-9)<br>TRIP-COOLER10-042623 (L39228-10)<br>TRIP-COOLER11-042623 (L39228-11) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD: RPDs ok<br>Field Duplicate:<br>BL5-WAT-BULK-01-042423<br>BL5-WAT-BULK-02-042423 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | A reduced sample volume was used for samples RC23-WAT-BULK-01-042423, RC12-WAT-BULK-01-042423, RC13-WAT-BULK-01-042123, RC13-WAT-BULK-01-042423, and EP17-WAT-BULK-01-042423 (SGS AXYS ID: L39229-4, -5, -6, -7, and -8, respectively) due to high analyte concentrations. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**One PFBA result was rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/2/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86035 (WG85029)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were extracted six to nine days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+ and UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Four results was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW7Q-1-GW-64-74-01-012623 | L38792-15 | 13C2-PFTeDA | 20.9 | J+<br>PFTrDA: UJ |
| OW7Q-1-GW-64-74-01-012623 | L38792-15 | d9-NEt-FOSE | 4.89 | R |
| OW7Q-1-GW-64-74-01-012623 | L38792-15 | d7-NMe-FOSE | 16.1 | UJ |
| MW10A-GW-273-283-01-012623 | L38792-24 | 13C4-PFBA | 16.2 | J+ |
| MW10A-GW-273-283-01-012623 | L38792-24 | 13C2-8:2 FTS | 48.3 | UJ |
| MW10B-GW-130-140-01-012623 | L38792-25 | 13C4-PFBA | 18.0 | J+ |
| MW14A-GW-316-326-01-012723 | L38792-27 | 13C4-PFBA | 26.5 | J+ |
| MW14A-GW-316-326-01-012723 | L38792-27 | 13C2-8:2 FTS | 39.5 | UJ |
| MW14A-GW-316-326-01-012723 | L38792-27 | D3-MeFOSAA | 43.2 | UJ |
| MW18A-GW-352-362-01-012623 | L38792-31 | 13C4-PFBA | 20.2 | J+ |
| MW18B-GW-225-235-01-012623 | L38792-32 | 13C4-PFBA | 41.4 | J+ |
| MW20B-GW-90-100-01-012623 | L38792-34 i | 13C4-PFBA | 3.81 | R |
| OW20J-1-GW-310-320-01-012523 | L38792-36 | 13C4-PFBA | 10.4 | J+ |
| OW20P-1-GW-150-160-01-012523 | L38792-37 | 13C4-PFBA | 8.42 | R |
| OW20S-1-GW-190-200-01-012523 | L38792-38 | 13C4-PFBA | 10.3 | J+ |
| OW20T-1-GW-98-108-01-012523 | L38792-39 | 13C4-PFBA | 6.15 | R |
| OW20S-2-GW-200-210-01-012523 | L38792-40 | 13C4-PFBA | 22.7 | J+ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **OW7Q-1-GW-64-74-03-012623:** | | | | | |
| PFBA | **135 /** 125 | 70-130 | 0.708 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **144 / 144** | 70-130 | 0.322 | 30 | |
| PFDoS | **57.3 / 51.8** | 70-130 | 8.55 | 30 | The results for samples OW7Q-1-GW-64-74-01-012623, OW7Q-1-GW-64-74-02-012623 and OW7Q-1-GW-64-74-03-012623 were qualified as estimated biased low (J-). |
| 11Cl-PF3OUdS | **60.0 / 61.7** | 70-130 | 4.36 | 30 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW7Q-1-GW-64-74-01-012623 | L38792-15 | PFTeDA |
| MW10A-GW-273-283-01-012623 | L38792-24 | PFOS |
| MW10B-GW-130-140-01-012623 | L38792-25 | PFOS |
| MW10C-GW-10-20-01-012623 | L38792-26 | PFOS |
| MW18A-GW-352-362-01-012623 | L38792-31 | PFOS |
| MW18B-GW-225-235-01-012623 | L38792-32 | PFOS |
| MW20B-GW-90-100-01-012623 | L38792-34 i | PFOS |
| OW20J-1-GW-310-320-01-012523 | L38792-36 | PFDA |
| OW20J-1-GW-310-320-01-012523 | L38792-36 | PFNA |
| OW20P-1-GW-150-160-01-012523 | L38792-37 i | PFOS |
| OW20S-1-GW-190-200-01-012523 | L38792-38 | PFOS |
| OW20S-1-GW-190-200-01-012523 | L38792-38 | PFDA |
| OW20T-1-GW-98-108-01-012523 | L38792-39 | PFOS |
| OW20S-2-GW-200-210-01-012523 | L38792-40 | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for sample MW14D-GW-16-21-01-012423 (L38792-35) was reported from a five times dilution analysis due to a high analyte concentration.

The samples and analytes listed below were re-analyzed due to carry-over. Results were reported from the re-analysis.

| Sample ID | Analytes |
|---|---|
| MW10A-GW-273-283-01-012623 (L38792-24) | PFHxA and PFHpA |
| MW20B-GW-90-100-01-012623 (L38792-34) | PFBA and PFOS |
| OW20P-1-GW-150-160-01-012523 (L38792-37) | PFOA and PFOS |

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG86035 (WG85029) Report date (mm/dd/yyyy): 6/22/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/2/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were received within hold time.<br><br>See data qualifications summary (attachment) for hold time exceedances and qualifications. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature and hold time exceedances are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER1-013023 (L38792-1)<br>TRIP-COOLER2-013023 (L38792-2)<br>TRIP-COOLER3-013023 (L38792-3)<br>TRIP-COOLER4-013023 (L38792-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OW7Q-1-GW-64-74-03-012623 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: RPDs ok<br>Field Triplicate:<br>OW7Q-1-GW-64-74-01-012623<br>OW7Q-1-GW-64-74-02-012623<br>OW7Q-1-GW-64-74-03-012623 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Four results were rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/2/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86155 (WG85030)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+ and UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Six results was rejected (R) due to a severely low labeled compound recovery. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW20S-3-GW-165-175-01-012523 | L38792-41 | 13C2-8:2 FTS | 49.7 | UJ |
| OW20S-3-GW-165-175-01-012523 | L38792-41 | 13C4-PFBA | 1.57 | R |
| OW20S-3-GW-165-175-01-012523 | L38792-41 | 13C5-PFPeA | 20.0 | J+ |
| MW21A-GW-410-420-01-012623 | L38792-42 | 13C4-PFBA | 6.89 | R |
| MW22B-GW-290-300-01-012623 | L38792-45 | 13C4-PFBA | 42.2 | J+ |
| MW22C-GW-160-170-01-012623 | L38792-46 | 13C4-PFBA | 40.2 | J+ |
| MW26A-GW-380-390-01-012623 | L38792-47 | 13C4-PFBA | 1.82 | R |
| MW26A-GW-380-390-01-012623 | L38792-47 | 13C5-PFPeA | 15.1 | J+ |
| MW26C-GW-130-140-01-012623 | L38792-49 | 13C4-PFBA | 4.49 | R |
| MW27A-GW-221-222-01-012623 | L38792-50 | 13C4-PFBA | 5.95 | R |
| MW27B-GW-120-130-01-012623 | L38792-51 | 13C4-PFBA | 5.01 | R |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW8B-GW-50-60-03-012523:** | | | | | |
| PFBA | **146** / 100 | 70-130 | 2.54 | 30 | The sample results were greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **142 / 135** | 70-130 | 0.621 | 30 | |
| PFOS | **197** / 116 | 70-130 | 6.08 | 30 | |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW20J-3-GW-280-290-01-012523 | L38792-41 | PFNS |
| OW20J-3-GW-280-290-01-012523 | L38792-41 | PFOS |
| OW20S-3-GW-165-175-01-012523 | L38792-42 | PFNS |
| OW20S-3-GW-165-175-01-012523 | L38792-42 | PFOS |
| MW22B-GW-290-300-01-012623 | L38792-45 | PFDA |
| MW22B-GW-290-300-01-012623 | L38792-45 | PFNA |
| MW26A-GW-380-390-01-012623 | L38792-47 | PFOS |
| MW26B-GW-249-259-01-012623 | L38792-48 | PFNS |
| MW26B-GW-249-259-01-012623 | L38792-48 | PFOS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW26C-GW-130-140-01-012623 | L38792-49 | PFOS |
| MW27A-GW-221-222-01-012623 | L38792-50 | PFNS |
| MW27B-GW-120-130-01-012623 | L38792-51 | PFOS |
| W6201-GW-109-124-01-012523 | L38792-53 | PFNS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

The PFOS result for sample MW22A-GW-429-439-01-012623 (L38792-44) was reported from a five times dilution analysis due to a high analyte concentration.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG86155 (WG85030) Report date (mm/dd/yyyy): 6/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/5/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER2-013023 (L38792-2)<br>TRIP-COOLER4-013023 (L38792-4)<br>TRIP-COOLER5-013023 (L38792-5) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW8B-GW-50-60-03-012523 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD: RPDs ok<br>Field Triplicate:<br>MW8B-GW-50-60-01-012523<br>MW8B-GW-50-60-02-012523<br>MW8B-GW-50-60-03-012523 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | A reduced sample volume was used for sample MW21A-GW-410-420-01-012623 (L38792-43) due to high solids. |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

**Six results were rejected (R) due to a severely low labeled compound recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/5/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86274 (WG85780)**

**Chain of custody preservation, and holding time (Section 1C):** The sample was received at a temperature of 7.3 °C, and was above the < 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C.

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85780-102 (A) | NFDHA | 132 | 70-130 | Associated results were nondetect, and were acceptable without qualification. |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZID-GW-22-27-01-041923 | L39228-90 R | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG86274 (WG85780) Report date (mm/dd/yyyy): 7/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/13/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | The sample was received at a temperature of 7.3 °C, and was above the < 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER8-042623 (L39228-8) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD: RPDs ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/13/2023

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG87556 (WG86674)**

**Chain of custody preservation, and holding time (Section 1C):** Some of the samples were received at temperatures greater than the 6 °C criteria at 6.7°C, 7.4°C, 8.0°C, 8.9°C, 10.0°C, 10.1°C, and 11.2°C. Detects associated with receipt temperatures above 10°C were qualified as estimated biased low (J-), and the reporting limit for nondetects were qualified as estimated (UJ).

| Sample ID | Lab ID |
|---|---|
| TRIP-COOLER1-062123 | L39586-1 |
| EQ-DRILLWAT-052523 | L39586-2 |
| TRIP-COOLER4-072523 | L39828-4 |

Sample EQ-DRILLWAT-052523 (L39586-2) was extracted 22 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/14/23 19:11 | ADONA | 140% | 70-130% | ADONA results were nondetect, and were acceptable without qualification. |

**Trip blank and equipment blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified BJ.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| EQ-DRILLWAT-052523 | PFBS<br>PFHxS<br>PFBA<br>PFPeA<br>PFHxA<br>PFOA<br>PFOSA | 0.738 J<br>0.582 J<br>128<br>3.04 J<br>1.06 J<br>0.84 J<br>0.792 J | Associated samples were not reported in this SDG. |
| TRIP-COOLER1-072523 | PFOSA | 0.423 J | Associated samples were not reported in this SDG. |
| TRIP-COOLER2-072523 | PFOSA | 0.545 J | |
| TRIP-COOLER4-072523 | PFOSA | 0.69 J | |
| TRIP-COOLER6-072523 | PFOSA | 0.642 J | |
| TRIP-COOLER1-080223 | PFOSA | 0.847 J | |
| EQ-DRILLPUMP-062623 | PFOSA | 0.483 J | |
| TRIP-COOLER1-081423 | PFOSA | 0.443 J | Associated sample concentrations were within 10 times the blank concentration and qualified BJ:<br><br>EQ-BLANK-BUCKET-01-080823<br>WL18-WAT-BULK-01-080823<br>WL18-WAT-BULK-02-080823<br>WL19-WAT-BULK-01-080823<br>WL20-WAT-BULK-01-080823 |

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| TRIP-COOLER1-062123 | 6:2 FTS | 2.25 BJ | 6:2 FTS detects were determined to be laboratory contamination (see method blank qualifications). |
| EQ-DRILLWAT-052523 | | 2.45 BJ | |
| TRIP-COOLER1-072523 | | 2.19 BJ | |
| TRIP-COOLER2-072523 | | 2.47 BJ | |
| TRIP-COOLER3-072523 | | 2.51 BJ | |
| TRIP-COOLER4-072523 | | 2.36 BJ | |
| TRIP-COOLER5-072523 | | 2.4 BJ | |
| TRIP-COOLER6-072523 | | 2.5 BJ | |
| TRIP-COOLER7-072523 | | 2.69 BJ | |
| TRIP-COOLER1-080223 | | 2.31 BJ | |
| EQ-DRILLPUMP-062623 | | 2.12 BJ | |
| TRIP-COOLER1-081423 | | 3.44 BJ | |
| EQ-BLANK-BUCKET-01-080823 | | 3.22 BJ | |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified BJ.

| Method Blank ID | Compound | Blank Concentration | Results Qualified |
|---|---|---|---|
| WG86674-101 | 6:2 FTS | 2.59 ng/l | All samples in this SDG detected 6:2 FTS at concentrations within 10 times the blank concentration. The results were qualified BJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Detects associated with high recoveries were qualified as J- (unless previously qualified BJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery | Lab Recovery Limits | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|---|
| TRIP-COOLER4-072523 | L39828-4 | 13C8-PFOSA | 138 | 30-130 | J- |
| EQ-BLANK-BUCKET-01-080823 | L39903-2 | 13C8-PFOSA | 137 | 30-130 | No Qualifier |
| WL18-WAT-BULK-02-080823 | L39903-4 | 13C8-PFOSA | 133 | 30-130 | J- |
| WL19-WAT-BULK-01-080823 | L39903-5 | d9-NEt-FOSE | 152 | 10-150 | No Qualifier |
| WL19-WAT-BULK-01-080823 | L39903-5 | d7-NMe-FOSE | 151 | 10-150 | No Qualifier |
| WL19-WAT-BULK-01-080823 | L39903-5 | 13C8-PFOSA | 141 | 30-130 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG86674-102 | ADONA | **131/135** | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | NFDHA | **64.8**/75.0 | 70-130 | Associated results were nondetect and qualified as estimated (UJ):<br><br>All samples in this SDG were associated with the LCS. |

| LCS IDs | Compound | LCS/LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG86674-103 (Low Level) | 6:2 FTS | **139** | 70-130 | Associated results were previously qualified BJ due to laboratory contamination. No further qualifications were warranted. |
| | ADONA | **140** | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | PFOSA | **131** | 70-130 | Associated detections were qualified as estimated biased high (J+), unless previously qualified BJ:<br><br>EQ-DRILLWAT-052523 (L39586-2)<br>TRIP-COOLER1-072523 (L39828-1)<br>TRIP-COOLER2-072523 (L39828-2)<br>TRIP-COOLER4-072523 (L39828-4)<br>TRIP-COOLER6-072523 (L39828-6)<br>TRIP-COOLER1-080223 (L39867-1)<br>EQ-DRILLPUMP-062623 (L39867-2)<br>TRIP-COOLER1-081423 (L39903-1) |

**Bold** indicates an exceedance

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL19-WAT-BULK-01-080823 | L39903-5 | PFHpS |
| WL20-WAT-BULK-01-080823 | L39903-6 | PFBS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG87556 (WG86674) Report date (mm/dd/yyyy): 10/25/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 11/28/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Some of the samples were received at temperatures of above the 6 °C criteria. Results associated with receipt temperatures greater than 10 °C were qualified as listed in the data qualifications summary (attachment). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Ten trip blanks and three equipment blank samples were reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for a list of field and trip blank detections and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 2.59 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits listed in lab report used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD RPDs ok<br>Field Duplicate:<br>WL18-WAT-BULK-01-080823<br>WL18-WAT-BULK-02-080823 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/28/2023

**SGS AXYS DPWG87665** 3/1/24 **LS**

Initial Cal 3/1/23 **PFOS**

| Area Counts | | | | | |
|---|---|---|---|---|---|
| IS (3C8-PFOS) | PFOS | Conc Std (ng/mL) | RFF - calculated | RFF - Cal form | p 665 |
| | | | | | |
| 33252 | 1656 | 0.4 | 1.2528 | 1.25 | |
| 27581 | 2492 | 0.8 | 1.1364 | 1.14 | |
| 24873 | 5560 | 2 | 1.1246 | 1.12 | |
| 22908 | 11650 | 5 | 1.0234 | 1.02 | |
| 27517 | 30390 | 10 | 1.1113 | 1.11 | |
| 29103 | 67483 | 20 | 1.1666 | 1.17 | |
| 33802 | 185112 | 50 | 1.1021 | 1.10 | |
| 26294 | 692168 | 250 | 1.0595 | 1.06 | |
| | | Avg: | 1.1221 | 1.12 | 1.12210 |
| | | %RSD: | 6.1594 | 6.16 | Internal Std Conc: 10.062 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | Lab ID |
|---|---|---|---|---|---|---|---|---|---|
| MW23B-GW-299-309-01-060523 | PFOS | 7.67E+04 | 1.60E+04 | 1 | 636 | 636 | 0.0676 | 0.004 | L39589-6 |
| MW23A-GW-422-432-01-060223 | PFOS | 2.04E+04 | 1.97E+04 | 1 | 139 | 139 | 0.0667 | 0.004 | L39589-7 |
| WG86356-102 | PFOS | 1.05E+04 | 1.91E+04 | 1 | 4.93 | 4.94 | 1.00 | 0.004 | |
| WG86356-103 | PFOS | 3.50E+03 | 1.99E+04 | 1 | 1.58 | 1.58 | 1.00 | 0.004 | |

| Compound | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %R Reported |
|---|---|---|---|---|---|---|---|
| CCV 8/29/23 9:37 | 1.75E+04 | 3.38E+04 | 4.643 | 4.63 | 5.00 | 92.60 | 92.6% |
| CCV 8/29/23 13:17 | 1.74E+04 | 3.42E+04 | 4.562 | 4.57 | 5.00 | 91.40 | 91.4% |

| | | Raw Data (ng/L) | Reported (ng/L) | |
|---|---|---|---|---|
| Method Blank ( WG86356-101) | PFOS | 0.067 | <0.4 | verified <0.4 |

# Data Validation Report

| | | |
|---|---|---|
| Project: | MPCA – Project 1007 | |
| Laboratory: | SGS AXYS – Sidney, BC, Canada | |
| Data Package: | DPWG87665 (Analysis WG86356) dated 12/15/2023 | |
| Analyses/Method: | SGS AXYS Method MLA-110 / 1633 | |
| Validation Level: | Stage 4 | |
| Prepared by: | Lisa Smith (CEAC) | Completed on: 3/4/2024 |

The groundwater samples listed below were collected at the Project 1007 Site on various dates from May 25 to July 28, 2023.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| **Groundwater Samples:** | | | |
| MW23C-GW-151-161-01-060223 | | 6/2/2023 | L39589-1 |
| MW23C-GW-56-60-02-052523 | Field Duplicate of MW23C-GW-56-60-01-052523 | 5/25/2023 | L39589-2 |
| MW23C-GW-56-60-01-052523 | | 5/25/2023 | L39589-3 |
| MW23C-GW-56-60-03-052523 | | 5/25/2023 | L39589-4 (A) |
| MW23C-GW-106-110-01-052523 | | 5/25/2023 | L39589-5 |
| MW23B-GW-299-309-01-060523 | | 6/5/2023 | L39589-6 |
| MW23A-GW-422-432-01-060223 | | 6/2/2023 | L39589-7 |
| MW23A-GW-422-432-01-071923 | | 7/19/2023 | L39828-23 |
| MW23B-GW-299-309-01-071923 | | 7/19/2023 | L39828-24 |
| MW23C-GW-151-161-01-071923 | | 7/19/2023 | L39828-25 |
| MW25A-GW-390-400-01-071923 | | 7/19/2023 | L39828-68 |
| MW25B-GW-260-270-01-071923 | | 7/19/2023 | L39828-69 |
| MW21A-GW-410-420-01-072823 | | 7/28/2023 | L39866-1 |
| MW25B-GW-26-30-01-062623 | | 6/26/2023 | L39866-2 |
| MW25B-GW-76-80-01-062723 | | 6/27/2023 | L39866-3 |

Data validation activities were conducted with reference to:

- *Guidance for Per- and Polyfluoroalkyl Substances: Analytical, Minnesota Pollution Control Agency (MPCA), October 2023.*
- *Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.*

Control limits used for this review were as indicated in the guidance documents listed above, and the analytical method (MLA-110 / 1633).

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

✓ Data completeness (chain-of-custody (COC)/sample integrity
✗ Sample Receipt and Holding times
✓ Initial calibration/continuing calibration verification (CCVs)
✗ Laboratory and trip blanks
✗ Labeled Compounds
✗ Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓ Laboratory control samples (LCSs)
✓ Field duplicates
✗ Quantitation and Result Verifications

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Thirteen 6:2 FTS results were qualified BJ due to laboratory contamination and four PFOSA results were qualified BJ due to trip contamination. In addition, results were qualified as estimated (J) with bias flags (+/-) as appropriate, due to sample receipt temperatures, holding times, labeled compounds, ion ratio, and MS/MSDs. Result qualified are listed in the Table 1 Data Validation Summary of Qualified Data, and discussed in detail in the following section. Data validation qualifiers override any assigned laboratory data flags.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete.

**Sample Receipt and Holding Times**

The samples were received at the laboratory intact; however, some of the samples were above the 6 °C temperature requirement and received at 6.7 °C and 10.1 °C. Results associated with temperature receipts greater than 10 °C, were qualified as estimated (UJ/J-).

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. The samples listed below were extracted over the 90-day hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

| Sample ID | Lab ID |
|---|---|
| MW23C-GW-56-60-02-052523 | L39589-2 |
| MW23C-GW-56-60-01-052523 | L39589-3 |
| MW23C-GW-56-60-03-052523 | L39589-4 (A) |
| MW23C-GW-106-110-01-052523 | L39589-5 |

**Calibration**

Initial calibrations for the target compounds were performed using response factor (RF) calibrations. Relative standard deviations (RSDs) of the RFs were within 20%.

The low calibration point was at or below the reporting limit. In addition, the low calibration standard was within ± 50% of the true value. Other calibration points were within ± 30% of the true value.

Primary transitions specified in the MPCA guidance document were used during calibration and sample quantitation. Some of the secondary transitions listed in the MPCA guidance document were not used, but the secondary transitions followed Method 1633 requirements.

Second source verification recoveries were within ± 30% of the true value.

Continuing calibration verifications (CCVs) were performed at the correct frequency. CCVs were within the criteria of 70-130% recovery.

Two CCVs were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Trip Blanks**

Method blanks were analyzed to assess laboratory contamination. 6:2 FTS was detected in the method blank at a concentration of 2.10 J ng/l. The case narrative indicated that an interferent eluted closely with this analyte. 6:2 FTS sample concentrations within 10 times the blank concentration were qualified BJ.

Trip blanks are used to assess contamination that occurs during sample shipping. Four trip blank were associated with the sample data; however, the results were not reported in this SDG. The trip blank detects are summarized below.

| Trip Blank ID | Sample Date | Compound | Units | Trip Blank Results | Qualifiers |
|---|---|---|---|---|---|
| TRIP-COOLER1-062123 | 6/21/2023 | 6:2 FTS | ng/L | 2.25 BJ | Laboratory Contamination |
| TRIP-COOLER2-072523 | 7/25/2023 | 6:2 FTS | ng/L | 2.47 BJ | Laboratory Contamination |
| | 7/25/2023 | PFOSA | ng/L | 0.545 J+ | Sample detects within 10 times the blank concentration were qualified BJ:<br><br>MW23A-GW-422-432-01-071923 (L39828-23)<br>MW23B-GW-299-309-01-071923 (L39828-24) |
| TRIP-COOLER7-072523 | 7/25/2023 | 6:2 FTS | ng/L | 2.69 BJ | Laboratory Contamination |
| TRIP-COOLER1-080223 | 8/2/2023 | 6:2 FTS | ng/L | 2.31 BJ | Laboratory Contamination |
| | 8/2/2023 | PFOSA | ng/L | 0.847 J+ | Sample detects within 10 times the blank concentration were qualified BJ:<br><br>MW25B-GW-26-30-01-062623 (L39866-2)<br>MW25B-GW-76-80-01-062723 (L39866-3) |

**Labeled Compounds**

Labeled compounds are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within method limits of 50% to 150%, with the exception of the results listed below. Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW23C-GW-56-60-01-052523 | L39589-3 | 13C2-PFTeDA | 22.9 | UJ<br>PFTrDA: UJ |
| MW23C-GW-56-60-01-052523 | L39589-3 | d9-NEt-FOSE | 29.9 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW23C-GW-56-60-01-052523 | L39589-3 | d7-NMe-FOSE | 45.1 | UJ |
| MW23B-GW-299-309-01-071923 | L39828-24 | d7-NMe-FOSE | 159 | No Qualifier |
| MW25B-GW-26-30-01-062623 | L39866-2 | 13C2-PFTeDA | 33.6 | UJ<br>PFTrDA: UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | 13C2-PFTeDA | 12.7 | UJ<br>PFTrDA: UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | 13C2-PFDoA | 47.0 | UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | 13C8-PFOS | 37.6 | UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | D3-N-MeFOSA | 49.1 | UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | D5-N-EtFOSA | 45.6 | UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | d7-NMe-FOSE | 15.9 | UJ |
| MW25B-GW-76-80-01-062723 | L39866-3 | d7-NMe-FOSE | 28.7 | UJ |

## MS/MSD Results

Matrix spikes are analyzed to determine the effects of sample matrix on the measurement methodology. MS/MSD analysis was performed on sample MW23C-GW-56-60-03-052523, and recoveries and relative percent differences (RPDs) were with MPCA limits, except as summarized below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW23C-GW-56-60-03-052523:** | | | | | |
| 11Cl-PF3OUdS | 72.5 **/ 61.9** | 70-130 | 15.2 | 30 | The results for samples MW23C-GW-56-60-01-052523, MW23C-GW-56-60-02-052523, and MW23C-GW-56-60-03-052523 were nondetect and qualified as estimated (UJ). |
| PFDoS | **58.5 / 45.4** | 70-130 | 24.7 | 30 | |

**Bold indicates an exceedance**

## LCS Results

LCSs are analyzed to monitor the accuracy and precision of the analytical method independent of matrix effects. The LCS %Rs were within the MPCA limits and were acceptable.

LCS results were recalculated for PFOS during validation. Results reported are verified to be correct.

## Field Duplicate Results

Samples MW23C-GW-56-60-02-052523 and MW23C-GW-56-60-03-052523 were collected to assess field precision for sample MW23C-GW-56-60-01-052523. RPDs were within the 30 percent limit for water sample, or results were within ± the reporting limit (RL). RPDs were within the 30 percent limit for water sample, or results were within ± the limit of quantification.

## Quantitation and Result Verifications

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review. Recalculations were performed for two PFOS results and recalculations are included in Attachment A. No anomalies were observed during the re-calculation and verification of the results.

Nine samples were analyzed using a reduced sample volume. The case narrative indicated that this was necessary due to high suspended solids, and to avoid clogging the SPE.

Quantitations were performed to include branched isomers where applicable. In addition, signal to noise ratios were greater than the method criteria of 3:1 (primary transition); and ion ratios were within 50% to 150% of the initial calibration ratios, except for results listed in the table below which did not meet criteria and are considered biased high (J+).

| Sample ID | Lab ID | Analyte | Reported as Estimated Maximum |
|---|---|---|---|
| MW23B-GW-299-309-01-071923 | L39828-24 | PFOA | No |
| MW23B-GW-299-309-01-071923 | L39828-24 | PFDA | Yes |
| MW23C-GW-151-161-01-071923 | L39828-25 | PFOS | Yes |
| MW25A-GW-390-400-01-071923 | L39828-68 | PFPeS | Yes |
| MW21A-GW-410-420-01-072823 | L39866-1 | PFNS | Yes |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the limit of quantitation were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW23C-GW-56-60-02-052523 (L39589-2)<br>MW23C-GW-56-60-01-052523 (L39589-3)<br>MW23C-GW-56-60-03-052523 (L39589-4 (A))<br>MW23C-GW-106-110-01-052523 (L39589-5) | All PFAS | ng/L | Detects: J-<br>Nondetects: UJ | ht,sr |
| MW23C-GW-151-161-01-060223 (L39589-1)<br>MW23B-GW-299-309-01-060523 (L39589-6)<br>MW23A-GW-422-432-01-060223 (L39589-7) | All PFAS | ng/L | Detects: J-<br>Nondetects: UJ | sr |
| MW23C-GW-151-161-01-060223 (L39589-1)<br>MW23C-GW-56-60-02-052523 (L39589-2)<br>MW23C-GW-56-60-01-052523 (L39589-3)<br>MW23C-GW-56-60-03-052523 (L39589-4 (A))<br>MW23C-GW-106-110-01-052523 (L39589-5)<br>MW23A-GW-422-432-01-071923 (L39828-23)<br>MW23B-GW-299-309-01-071923 (L39828-24)<br>MW23C-GW-151-161-01-071923 (L39828-25)<br>MW25A-GW-390-400-01-071923 (L39828-68)<br>MW25B-GW-260-270-01-071923 (L39828-69)<br>MW21A-GW-410-420-01-072823 (L39866-1)<br>MW25B-GW-26-30-01-062623 (L39866-2)<br>MW25B-GW-76-80-01-062723 (L39866-3) | 6:2 FTS | ng/L | BJ | mb |
| MW23A-GW-422-432-01-071923 (L39828-23)<br>MW23B-GW-299-309-01-071923 (L39828-24)<br>MW25B-GW-26-30-01-062623 (L39866-2)<br>MW25B-GW-76-80-01-062723 (L39866-3) | PFOSA | ng/L | BJ | tb |
| MW23C-GW-56-60-01-052523 (L39589-3)<br>MW25B-GW-26-30-01-062623 (L39866-2)<br>MW25B-GW-76-80-01-062723 (L39866-3) | PFTeDA<br>PFTrDA | ng/L | UJ | s |
| MW23C-GW-56-60-01-052523 (L39589-3)<br>MW25B-GW-76-80-01-062723 (L39866-3) | N-MeFOSE<br>N-EtFOSE | ng/L | UJ | s |
| MW23B-GW-299-309-01-071923 (L39828-24) | N-MeFOSE | ng/L | UJ | s |
| MW25B-GW-76-80-01-062723 (L39866-3) | PFDoA<br>PFOS<br>N-MeFOSA<br>N-EtFOSA | ng/L | UJ | s |

| Sample ID | Analyte | Units | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|
| MW23C-GW-56-60-02-052523 (L39589-2)<br>MW23C-GW-56-60-01-052523 (L39589-3)<br>MW23C-GW-56-60-03-052523 (L39589-4 (A)) | 11Cl-PF3OUdS<br>PFDoS | ng/L | UJ | ms |
| MW23B-GW-299-309-01-071923 (L39828-24)<br>MW23C-GW-151-161-01-071923 (L39828-25)<br>MW25A-GW-390-400-01-071923 (L39828-68)<br>MW21A-GW-410-420-01-072823 (L39866-1) | PFDA<br>PFOS<br>PFPeS<br>PFNS | ng/L | J+ | em |
| MW23B-GW-299-309-01-071923 (L39828-24) | PFOA | ng/L | J+ | q |

(1): Data Validation Qualifiers:

| | |
|---|---|
| BJ | Laboratory or trip contamination |
| J | Estimated (+/- indicates high or low bias) |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| ht | Hold time |
| em | Estimated maximum |
| mb | Method blank |
| ms | Matrix spike |
| q | Quantitation (ion ratios) |
| sr | Sample receipt |
| s | Labeled compound |
| tb | Trip blank |

**Attachment A**

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG88710 (WG88283)**

**Chain of custody preservation, and holding time (Section 1C):** Eight of the samples were received at temperatures greater than the 6 °C criteria at 9.1°C. Results were not qualified, as the samples were received at temperatures < 10 °C.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/9/24 12:25 | 7:3 FTCA | 132% | 70-130% | The results were nondetect, and were acceptable without qualification. |
| CCV 1/9/24 15:50 | 7:3 FTCA | 136% | 70-130% | The results were nondetect, and were acceptable without qualification. |
| | NFDHA | 133% | 70-130% | |
| CCV 1/9/24 18:24 | 7:3 FTCA | 136% | 70-130% | The results were nondetect, and were acceptable without qualification. |

**Trip blank and equipment blank detections (Section 3Aii):** Associated trip blank detections are listed in the table below (these trip blanks were not reported in this SDG). Associated sample detects within 10 times the blank concentrations were qualified BJ.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| TRIP-COOLER4-110123 | PFOS | 0.726 J | Associated sample concentrations were within 10 times the blank concentration and qualified BJ:<br><br>MW25B-GW-260-270-01-102623 (L40481-45)<br>MW26B-GW-249-259-01-102623 (L40481-47)<br>MW26C-GW-130-140-01-102623 (L40481-48) |
| TRIP-COOLER5-110123 | PFOS | 2.4 | Associated sample concentrations were within 10 times the blank concentration and qualified BJ:<br><br>PZGS-GW-20-30-01-102523 (L40481-54)<br>PZH-1-GW-90-100-01-103023 (L40481-56) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. Four PFBA results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW23B-GW-299-309-01-102623 | L40481-42 | 13C4-PFBA | 5.83 | R |
| MW23C-GW-151-161-01-102623 | L40481-43 | 13C4-PFBA | 28.8 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW25A-GW-390-400-01-102623 | L40481-44 | 13C4-PFBA | 18.4 | J+ |
| MW25B-GW-260-270-01-102623 | L40481-45 | 13C4-PFBA | 5.35 | R |
| MW26A-GW-380-390-01-102623 | L40481-46 | 13C4-PFBA | 5.96 | R |
| MW26B-GW-249-259-01-102623 | L40481-47 | 13C4-PFBA | 11.6 | J+ |
| MW26C-GW-130-140-01-102623 | L40481-48 | 13C4-PFBA | 13.1 | J+ |
| PZGD-GW-35-40-01-102523 | L40481-51 | 13C4-PFBA | 17.8 | J+ |
| PZGD-GW-35-40-02-102523 | L40481-52 | 13C4-PFBA | 36.2 | J+ |
| PZGD-GW-35-40-02-102523 | L40481-52 | d9-NEt-FOSE | 153 | No Qualifier |
| PZGD-GW-35-40-03-102523 | L40481-53 (A) | 13C4-PFBA | 39.7 | J+ |
| PZGS-GW-20-30-01-102523 | L40481-54 | 13C4-PFBA | 47.7 | J+ |
| PZHD-GW-21-26-01-102523 | L40481-55 | 13C4-PFBA | 24.9 | J+ |
| PZH-1-GW-90-100-01-103023 | L40481-56 | D5-EtFOSAA | 39.9 | UJ |
| PZH-1-GW-90-100-01-103023 | L40481-56 | D3-MeFOSAA | 42.8 | UJ |
| PZH-1-GW-90-100-01-103023 | L40481-56 | 13C2-PFTeDA | 46.6 | UJ<br>PFTrDA: UJ |
| BL5-WAT-BULK-01-102323 | L40481-57 | d9-NEt-FOSE | 25.0 | UJ |
| BL5-WAT-BULK-01-102323 | L40481-57 | d7-NMe-FOSE | 41.0 | UJ |
| BL5-WAT-BULK-01-102323 | L40481-57 | 13C4-PFBA | 9.95 | R |
| BL5-WAT-BULK-01-102323 | L40481-57 | 13C2-PFTeDA | 26.2 | UJ<br>PFTrDA: UJ |
| BL5-WAT-BULK-02-102323 | L40481-58 | 13C4-PFBA | 21.6 | J+ |
| BL5-WAT-BULK-02-102323 | L40481-58 | 13C2-PFTeDA | 44.3 | UJ<br>PFTrDA: UJ |
| EP16-WAT-BULK-01-102323 | L40481-59 | d9-NEt-FOSE | 18.7 | UJ |
| EP16-WAT-BULK-01-102323 | L40481-59 | d7-NMe-FOSE | 35.7 | UJ |
| EP16-WAT-BULK-01-102323 | L40481-59 | 13C4-PFBA | 28.6 | J+ |
| EP16-WAT-BULK-01-102323 | L40481-59 | 13C2-PFTeDA | 21.9 | UJ<br>PFTrDA: UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG88283-102 | PFEESA | 131 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | NFDHA | 145 | 70-130 | |
| WG88283-103 (Low Level) | 3:3 FTCA | 132 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | 5:3 FTCA | 134 | 70-130 | |
| | 7:3 FTCA | 138 | 70-130 | |
| | PFEESA | 143 | 70-130 | |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFOA | 132 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples except PZH-1-GW-90-100-01-103023 (L40481-56) |
| | PFOSA | 140 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>PZED-GW-16-21-02-102523 (L40481-50)<br>PZGD-GW-35-40-01-102523 (L40481-51)<br>PZGD-GW-35-40-02-102523 (L40481-52)<br>PZGD-GW-35-40-03-102523 (L40481-53 (A))<br>PZGS-GW-20-30-01-102523 (L40481-54)<br>BL5-WAT-BULK-01-102323 (L40481-57)<br>BL5-WAT-BULK-02-102323 (L40481-58)<br>EP16-WAT-BULK-01-102323 (L40481-59) |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **PZGD-GW-35-40-03-102523:** | | | | | |
| 7:3 FTCA | **135 / 135** | 70-130 | 6.51 | 30 | The results for samples PZGD-GW-35-40-01-102523, PZGD-GW-35-40-02-102523, and PZGD-GW-35-40-03-102523 were nondetect and were acceptable without qualification. |
| PFEESA | 130 / **135** | 70-130 | 3.42 | 30 | |
| PFBA | 99,2 **/ 131** | 70-130 | 3.30 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **143 / 171** | 70-130 | 1.39 | 30 | |
| PFOS | 73.1 **/ 152** | 70-130 | 5.00 | 30 | |

**Bold indicates an exceedance**

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below:

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%) |
|---|---|---|---|
| **BL5-WAT-BULK-01-102323 / BL5-WAT-BULK-02-102323:** | | | |
| PFMPA | 2.48 J | 4.52 | > ± 2xRL |

**Method detection limits/Report limits (Section 7B):**

Reduced sample volumes were used for samples MW23A-GW-422-432-01-102623 (L40481-41) and PZH-1-GW-90-100-01-103023 (L40481-56) due to high solids.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J), unless previously qualified due to blanks:

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW23C-GW-151-161-01-102623 | L40481-43 | PFOA |
| MW25B-GW-260-270-01-102623 | L40481-45 | PFHxA |
| MW25B-GW-260-270-01-102623 | L40481-45 | PFOS |
| MW26A-GW-380-390-01-102623 | L40481-46 | PFOS |
| MW26B-GW-249-259-01-102623 | L40481-47 | PFOS |
| MW26C-GW-130-140-01-102623 | L40481-48 | PFOS |
| PZGS-GW-20-30-01-102523 | L40481-54 | PFOS |
| PZH-1-GW-90-100-01-103023 | L40481-56 | PFOS |

The PFOS result for sample PZHD-GW-21-26-01-102523 (L40481-55) was reported from a dilution analysis due to a high sample concentration.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG88710 (WG88283) Report date (mm/dd/yyyy): 2/7/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/5/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Eight of the samples were received at temperatures of above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | Associated trip blanks were not reported in the SDG: |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for a list of trip blank detections and qualifications. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZGD-GW-35-40-03-102523 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field Duplicates:<br>PZED-GW-16-21-01-102523 / PZED-GW-16-21-02-102523<br>BL5-WAT-BULK-01-102323 / BL5-WAT-BULK-02-102323<br>Field triplicate:<br>PZGD-GW-35-40-01-102523 / PZGD-GW-35-40-02-102523 / PZGD-GW-35-40-03-102523 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>See data qualifications summary (attachment) for exceedances and qualifications. . |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for details. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

**Four PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/5/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG88932 (WG88779)**

**Chain of custody preservation, and holding time (Section 1C):** Samples were extracted 13 days over the 90 day hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances are listed below.

| LCS IDs | Compound | LCS / LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG88779-102 / WG88779-103 | PFEESA | 132 / 132 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| WG88779-104 | 6:2 FTS | 133 / -- | 70-130 | Associated results were nondetect and were acceptable without qualification. |

**Method detection limits/Report limits (Section 7B):**

Reduced sample volumes were used for samples reported in this SDG due to high solids.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG88932 (WG88779) Report date (mm/dd/yyyy): 2/12/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/20/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were recieived within hold time; however, were extracted 13 days over the 90 day hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | The associated trip blank was not reported in this SDG. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | TRIP-COOLER8-110123 |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☒ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | MS/MSDs not included in this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD RPDs ok<br>Field duplicates not included in this SDG. |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for details. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/20/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG88983 (WG88281)**

**Chain of custody preservation, and holding time (Section 1C):** Eleven of the samples were received at temperatures greater than the 6 °C criteria at 7.5°C, 9.1°C, and 10.1°C. Detects associated with receipt temperatures above 10°C were qualified as estimated biased low (J-), and the reporting limit for nondetects were qualified as estimated (UJ).

| Sample ID | Lab ID |
|---|---|
| TRIP-COOLER1-110123 | L40481-1 |
| MW2D-GW-7-17-01-102523 | L40481-12 |
| MW2D-GW-7-17-02-102523 | L40481-13 |
| MW2D-GW-7-17-03-102523 | L40481-14 (A) |
| MW2E-GW-80-90-01-102623 | L40481-15 |
| MW2F-GW-130-150-01-102523 | L40481-16 |
| MW6C-GW-35-40-01-102523 | L40481-17 |
| MW6C-GW-35-40-02-102523 | L40481-18 |
| MW6D-GW-8-18-01-102523 | L40481-19 |
| OW7S-1-GW-90-100-01-102623 | L40481-20 |

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/5/24 17:38 | NFDHA | 54.7% | 70-130% | Associated results were nondetect, and qualified as estimated (UJ):<br><br>TRIP-COOLER1-110123 (L40481-1)<br>TRIP-COOLER2-110123 (L40481-2)<br>TRIP-COOLER3-110123 (L40481-3)<br>TRIP-COOLER4-110123 (L40481-4)<br>TRIP-COOLER5-110123 (L40481-5)<br>TRIP-COOLER6-110123 (L40481-6)<br>TRIP-COOLER7-110123 (L40481-7)<br>TRIP-COOLER8-110123 (L40481-8) |
| CCV 1/5/24 21:01 | NFDHA | 64.2% | 70-130% | Associated results were nondetect, and qualified as estimated (UJ):<br><br>EQ-DIPPER-102623 (L40481-9)<br>EQ-PERI-102623 (L40481-10)<br>EQ-WHALE-102623 (L40481-11)<br>MW2D-GW-7-17-01-102523 (L40481-12)<br>MW2D-GW-7-17-02-102523 (L40481-13)<br>MW6C-GW-35-40-01-102523 (L40481-17)<br>MW6C-GW-35-40-02-102523 (L40481-18)<br>MW6D-GW-8-18-01-102523 (L40481-19)<br>OW7S-1-GW-90-100-01-102623 (L40481-20) |

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/5/24 23:30 | NFDHA | 52.7% | 70-130% | Associated results were nondetect, and qualified as estimated (UJ):<br><br>MW2D-GW-7-17-03-102523 (L40481-14 (A))<br>MW2E-GW-80-90-01-102623 (L40481-15)<br>MW2F-GW-130-150-01-102523 (L40481-16)<br>MW6C-GW-35-40-02-102523 (L40481-18)<br>MW6D-GW-8-18-01-102523 (L40481-19)<br>OW7S-1-GW-90-100-01-102623 (L40481-20) |
| CCV 1/6/24 2:00 | ADONA | 155% | 70-130% | The results were nondetect, and were acceptable without qualification. |
| | NFDHA | 61.7% | 70-130% | Closing CCV. |

**Trip blank and equipment blank detections (Section 3Aii):** Trip blank detections are listed in the table below. Equipment blanks were nondetect.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| TRIP-COOLER4-110123 | PFOS | 0.726 J | Associated samples were reported in SDGs DPWG88710 and DPWG89086. |
| TRIP-COOLER5-110123 | PFOS | 2.4 | Associated samples were reported in SDG DPWG88710. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2D-GW-7-17-03-102523 | L40481-14 (A) | 13C2-PFTeDA | 49.7 | UJ<br>PFTrDA: UJ |
| MW2E-GW-80-90-01-102623 | L40481-15 | 13C4-PFBA | 45.1 | J+ |
| MW6C-GW-35-40-01-102523 | L40481-17 | 13C4-PFBA | 48.0 | J+ |
| MW6D-GW-8-18-01-102523 | L40481-19 | 13C4-PFBA | 30.7 | J+ |
| OW7S-1-GW-90-100-01-102623 | L40481-20 | 13C4-PFBA | 10.6 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG88281-102 | ADONA | 149 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | NFDHA | 55.9 | 70-130 | The results were nondetect and were qualified as estimated (UJ). |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG88281-103 | ADONA | 154 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | NFDHA | 62.0 | 70-130 | The results were nondetect and were qualified as estimated (UJ). |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **MW2D-GW-7-17-03-102523:** | | | | | |
| NFDHA | **59.2 / 61.0** | 70-130 | 3.79 | 30 | The results for samples MW2D-GW-7-17-01-102523, MW2D-GW-7-17-02-102523, and MW2D-GW-7-17-03-102523 were nondetect and qualified as estimated (UJ). |
| PFBA | **138 / 135** | 70-130 | 0.234 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **225 / NC** | 70-130 | 8.49 | 30 | |
| ADONA | **150 / 145** | 70-130 | 3.15 | 30 | The results for samples MW2D-GW-7-17-01-102523, MW2D-GW-7-17-02-102523, and MW2D-GW-7-17-03-102523 were nondetect and were acceptable without qualification. |

**Bold indicates an exceedance**

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW6C-GW-35-40-01-102523 | L40481-17 | PFDA |
| OW7S-1-GW-90-100-01-102623 | L40481-20 | PFOS |
| OW7S-1-GW-90-100-01-102623 | L40481-20 | PFNA |

The PFOS result for sample MW2F-GW-130-150-01-102523 (L40481-16) was reported from a 5 times dilution analysis due to a high sample concentration.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

Detects with both high and low bias were qualified as estimated (J).

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG88983 (WG88281) Report date (mm/dd/yyyy): 2/15/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Eleven samples were received at temperatures of above the 6 °C criteria. Results received at temperatures > 10 °C were qualfied as listed in the attached data qualification summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blanks:<br>TRIP-COOLER1-110123 (L40481-1)<br>TRIP-COOLER2-110123 (L40481-2)<br>TRIP-COOLER3-110123 (L40481-3)<br>TRIP-COOLER4-110123 (L40481-4)<br>TRIP-COOLER5-110123 (L40481-5)<br>TRIP-COOLER6-110123 (L40481-6)<br>TRIP-COOLER7-110123 (L40481-7)<br>TRIP-COOLER8-110123 (L40481-8)<br>Equipment blanks:<br>EQ-DIPPER-102623 (L40481-9)<br>EQ-PERI-102623 (L40481-10)<br>EQ-WHALE-102623 (L40481-11) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | See the attached data qualification summary for detections. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | The associated samples were reported in SDGs DPWG88710 and DPWG89086. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2D-GW-7-17-03-102523 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field Duplicates:<br>MW6C-GW-35-40-01-102523 /<br>MW6C-GW-35-40-02-102523<br>Field triplicate:<br>MW2D-GW-7-17-01-102523 /<br>MW2D-GW-7-17-02-102523 /<br>MW2D-GW-7-17-03-102523 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89086 (WG88282)**

**Chain of custody preservation, and holding time (Section 1C):** Nine of the samples were received at temperatures greater than the 6 °C criteria at 7.5°C. Results were not qualified, as the samples were received at temperatures < 10 °C.

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/14/24 8:46 | 7:3 FTCA | 132% | 70-130% | Associated results were nondetect, and were acceptable without qualification. |
| CCV 1/14/24 10:54 | 11Cl-PF3OUdS | 135% | 70-130% | Associated results were nondetect, and were acceptable without qualification. |
| CCV 1/14/24 12:37 | 11Cl-PF3OUdS | 132% | 70-130% | Associated results were nondetect, and were acceptable without qualification. |

**Trip blank and equipment blank detections (Section 3Aii):** Trip blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| TRIP-COOLER4-110123 | PFOS | 0.726 J | Associated samples concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. Ten PFBA results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW7S-3-GW-100-110-01-102623 | L40481-21 | 13C4-PFBA | 18.5 | J+ |
| MW8A-GW-202-212-01-102623 | L40481-22 | 13C4-PFBA | 23.7 | J+ |
| MW8B-GW-50-60-01-102423 | L40481-23 | 13C4-PFBA | 45.0 | J+ |
| MW10A-GW-273-283-01-102523 | L40481-24 | 13C4-PFBA | 7.41 | R |
| MW10B-GW-130-140-01-102523 | L40481-25 | 13C4-PFBA | 11.3 | J+ |
| MW14A-GW-316-326-01-102523 | L40481-26 N2 | 13C2-8:2 FTS | 44.9 | UJ |
| MW14A-GW-316-326-01-102523 | L40481-26 N2 | 13C4-PFBA | 8.13 | R |
| MW20A-GW-130-140-01-102723 | L40481-30 | 13C4-PFBA | 5.90 | R |
| MW20B-GW-90-100-01-102723 | L40481-31 | 13C4-PFBA | 6.45 | R |
| OW20P-1-GW-150-160-01-102723 | L40481-32 | 13C4-PFBA | 11.3 | J+ |
| OW20S-1-GW-190-200-01- | L40481-33 | 13C4-PFBA | 4.69 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| 102723 | | | | |
| OW20S-3-GW-165-175-01-102723 | L40481-34 | 13C4-PFBA | 9.38 | R |
| OW20J-1-GW-310-320-01-102723 | L40481-35 | 13C4-PFBA | 3.37 | R |
| OW20J-1-GW-310-320-01-102723 | L40481-35 | 13C5-PFPeA | 49.7 | J+ |
| OW20T-1-GW-98-108-01-102723 | L40481-36 | 13C4-PFBA | 3.87 | R |
| MW21A-GW-410-420-01-102623 | L40481-37 | 13C4-PFBA | 3.63 | R |
| MW22B-GW-290-300-01-102623 | L40481-39 | 13C4-PFBA | 2.60 | R |
| MW22B-GW-290-300-01-102623 | L40481-39 | 13C5-PFPeA | 40.3 | J+ |
| MW22C-GW-160-170-01-102623 | L40481-40 | 13C4-PFBA | 15.6 | J+ |

**Method detection limits/Report limits (Section 7B):**

Reduced sample volumes were used for samples MW14B-GW-60-70-01-102423 (L40481-27), MW14D-GW-6-21-01-102423 (L40481-28), MW17A-GW-230-240-01-102523 (L40481-29), and MW22A-GW-429-439-01-102623 (L40481-38) due to high solids.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW10A-GW-273-283-01-102523 | L40481-24 | PFOS |
| MW10A-GW-273-283-01-102523 | L40481-24 | PFPeS |
| MW10B-GW-130-140-01-102523 | L40481-25 | PFOS |
| MW17A-GW-230-240-01-102523 | L40481-29 | PFOS |
| OW20P-1-GW-150-160-01-102723 | L40481-32 | PFOS |
| OW20S-1-GW-190-200-01-102723 | L40481-33 | PFOS |
| OW20S-3-GW-165-175-01-102723 | L40481-34 | PFOS |
| OW20J-1-GW-310-320-01-102723 | L40481-35 | PFNA |
| OW20T-1-GW-98-108-01-102723 | L40481-36 | PFOS |
| MW21A-GW-410-420-01-102623 | L40481-37 | PFNA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89086 (WG88282) Report date (mm/dd/yyyy): 2/23/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Nine samples were received at temperatures of above the 6 °C criteria. Results were not qualified, as the samples were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | The associated trip blanks were not reported in this SDG.<br>TRIP-COOLER2-110123 (L40481-2)<br>TRIP-COOLER3-110123 (L40481-3)<br>TRIP-COOLER4-110123 (L40481-4) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | See the attached data qualification summary for detections. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Associated samples concentrations were greater than 10 times the blank concentration. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD RPDs ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for details. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

**Ten PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89110 (WG88284)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %Recovery | %Recovery Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/12/24 22:35 | 7:3 FTCA | 132% | 70-130% | Associated results were nondetect, and were acceptable without qualification. |
| CCV 1/13/24 1:22 | 7:3 FTCA | 133% | 70-130% | Associated results were nondetect, and were acceptable without qualification. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), and detects associated with high recoveries were qualified J-, as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP17-WAT-BULK-01-102323 | L40481-60 | 13C4-PFBA | 15.7 | J+ |
| EP21-WAT-BULK-01-102323 | L40481-61 | 13C4-PFBA | 19.0 | J+ |
| EP24-WAT-BULK-01-102323 | L40481-62 | 13C2-PFTeDA | 46.6 | UJ<br>PFTrDA: UJ |
| EP24-WAT-BULK-01-102323 | L40481-62 | 13C2-PFDoA | 47.8 | UJ |
| EP24-WAT-BULK-01-102323 | L40481-62 | D5-EtFOSAA | 44.3 | UJ |
| EP24-WAT-BULK-01-102323 | L40481-62 | D3-MeFOSAA | 45.5 | UJ |
| PL41-GW-72-82-01-102323 | L40481-64 | 13C4-PFBA | 37.6 | J+ |
| PL41-GW-72-82-01-102323 | L40481-64 | 13C2-PFTeDA | 47.9 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-01-102323 | L40481-64 | D5-EtFOSAA | 48.6 | UJ |
| PL41-GW-72-82-01-102323 | L40481-64 | D3-MeFOSAA | 48.6 | UJ |
| RC3-WAT-BULK-01-102323 | L40481-65 | 13C5-PFPeA | 173 | J- |
| RC3-WAT-BULK-01-102323 | L40481-65 | 13C8-PFOSA | 161 | J- |
| RC3-WAT-BULK-01-102323 | L40481-65 | D5-N-EtFOSA | 162 | No Qualifier |
| RC3-WAT-BULK-01-102323 | L40481-65 | D3-N-MeFOSA | 161 | No Qualifier |
| RC3-WAT-BULK-01-102323 | L40481-65 | d9-NEt-FOSE | 223 | No Qualifier |
| RC3-WAT-BULK-01-102323 | L40481-65 | d7-NMe-FOSE | 192 | No Qualifier |
| RC3-WAT-BULK-01-102323 | L40481-65 | 13C3-HFPO-DA | 155 | No Qualifier |
| RC5-WAT-BULK-02-102323 | L40481-67 | d9-NEt-FOSE | 153 | No Qualifier |
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C4-PFBA | 20.4 | J+ |
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C3-PFBS | 48.9 | J+ |
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C4-PFHpA | 49.2 | J+ |
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C2-PFTeDA | 46.2 | UJ<br>PFTrDA: UJ |
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C2-4:2 FTS | 43.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD4-WAT-BULK-01-102323 | L40481-72 | 13C2-8:2 FTS | 46.9 | UJ |
| OD4-WAT-BULK-01-102323 | L40481-72 | D5-EtFOSAA | 43.3 | UJ |
| OD4-WAT-BULK-01-102323 | L40481-72 | D3-MeFOSAA | 42.4 | UJ |
| W6201-GW-109-124-01-110623 | L40516-3 | 13C5-PFPeA | 170 | J- |
| W6201-GW-109-124-01-110623 | L40516-3 | d9-NEt-FOSE | 202 | No Qualifier |
| W6201-GW-109-124-01-110623 | L40516-3 | d7-NMe-FOSE | 173 | No Qualifier |
| W6201-GW-109-124-01-110623 | L40516-3 | 13C3-HFPO-DA | 153 | No Qualifier |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **RC5-WAT-BULK-03-102323:** | | | | | |
| PFOA | **201** / 110 | 70-130 | 5.26 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **209 / 49.1** | 70-130 | 3.25 | 30 | |
| NFDHA | 83.2 / 120 | 70-130 | **38.7** | 30 | The results for samples RC5-WAT-BULK-01-102323, RC5-WAT-BULK-02-102323, and RC5-WAT-BULK-03-102323 were nondetect and were acceptable without qualification. |

**Bold indicates an exceedance**

**Method detection limits/Report limits (Section 7B):**

Reduced sample volumes were used for ten samples due to high sample concentrations or high solids.

The PFOS result for sample EP17-WAT-BULK-01-102323 was reported from a 5x dilution analysis due to a high sample concentration.

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OD4-WAT-BULK-01-102323 | L40481-72 | PFNA |
| W6102-GW-93-105-01-110623 | L40516-2 | PFOS |
| W6102-GW-93-105-01-110623 | L40516-2 | N-EtFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89110 (WG88284) Report date (mm/dd/yyyy): 2/26/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/28/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances. Results did not require qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-110723<br>Associated trip blanks that were not reported in this SDG:<br>TRIP-COOLER6-110123<br>TRIP-COOLER7-110123<br>TRIP-COOLER8-110123 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | 6:2 FTS 136 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | Sample results were nondetect |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | RC5-WAT-BULK-03-102323 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Result did not require qualification. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Triplicate Samples:<br>RC5-WAT-BULK-01-102323 /<br>RC5-WAT-BULK-02-102323 / |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | RC5-WAT-BULK-03-102323 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for details. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/28/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89715(WG88958)**

**Blanks – (Section 3.c):** PFOS was detected in the equipment blank EQ-WHALETUBE-012224, but was not detected in the associated investigative samples. No qualifier is required.

**Matrix spike/ Matrix spike duplicates – (Section 6AV):** Results associated with high recoveries were qualified as estimated as (J+).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG88958-104 (MS) | **PFOA*** | **45.9%** | 70-130% | No qualifiers |
| WG88958-104 (MS) | **PFHpS** | **138%** | 70-130% | J+ |
| WG88958-105 (MSD) | **PFHpS** | **133%** | 70-130% | J+ |

* MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines.).

| | | |
|---|---|---|
| | | |
| | | |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89715 (WG88958) Report date (mm/dd/yyyy): 5/3/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/10/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | TRIP-COOLER1-012924<br>TRIP-COOLER2-012924<br>TRIP-COOLER3-012924<br>TRIP-COOLER4-012924<br>TRIP-COOLER5-013024<br>TRIP-COOLER6-013024<br>TRIP-COOLER7-013024<br>EQ-DIPPER-012224<br>EQ-PERITUBE-012224<br>EQ-PURELIFE-012624<br>EQ-WHALETUBE-012224 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects were more than the RL:<br>PFOS 0.740 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☒ | ☐ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample MW2F-GW-130-150-03-012324 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☒ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicates:<br>MW2D-GW-7-17-01-012324 / MW2D-GW-7-17-02-012324<br>Field Triplicate:<br>MW2F-GW-130-150-01-012324 / MW2F-GW-130-150-02-012324 / MW2F-GW-130-150-03-012324 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/10/2026

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89712 (WG85602) Report date (mm/dd/yyyy): 4/29/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/10/2026

## 1. Chain of custody, preservation, and holding times

| | Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample PZED-GW-16-21-03-012224 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>MW14B-GW-60-70-01-012424 / MW14B-GW-60-70-02-012424<br>Fiekd Triplicate:<br>PZED-GW-16-21-01-012224 / PZED-GW-16-21-02-012224 / PZED-GW-16-21-03-012224 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**One result was rejected (R) due to a severely low labeled analog (EIS) recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/10/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89712 (WG 88960)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 03/17/24 15:33 | NFDHA | **133%** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 03/17/24 07:10 | NFDHA | **144%** | 70-130% | |
| CCV 03/17/24 09:31 | NFDHA | **135%** | 70-130% | |

**Laboratory control spike/Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ and J-). Results associated with high recoveries were nondetects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG88960-102 | NFDHA | **147%** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG88960-103 | NFDHA | **135%** | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OW7S-1-GW-90-100-01-012524 | L40956-19 | 13C2-4:2 FTS | 154 | No Qualifier |
| MW10B-GW-130-140-01-012524 | L40956-24 | 13C4-PFBA | 4.78 | R |
| MW10B-GW-130-140-01-012524 | L40956-24 | No Qualifier | 165 | No Qualifier |
| MW14A-GW-316-326-01-012524 | L40956-25 | 13C4-PFBA | 15.0 | J+ |
| MW14A-GW-316-326-01-012524 | L40956-25 | 13C2-4:2 FTS | 187 | No Qualifier |
| MW20A-GW-130-140-01-012424 | L40956-30 | 13C4-PFBA | 16.3 | J+ |

**Matrix spike/ Matrix spike duplicates – (Section 6AV):** Results associated with high recoveries were nondetects and were acceptable without qualification. Results associated with high recoveries were qualified as estimated as (J+). MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines.).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG88960-104 (MS) | **PFOA** | **165%** | 70-130% | No qualifiers |
| WG88960-104 (MS) | **PFHpS** | **131%** | 70-130% | J+ |
| WG88960-105 (MSD) | **PFOA** | **179%** | 70-130% | No qualifiers |
| WG88960-105 (MSD) | **PFHpS** | **135%** | 70-130% | J+ |

**10. Additional comments about lab report:** For the results listed below, the results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW7S-1-GW-90-100-01-012524 | L40956-19 | PFOS |
| MW17A-GW-230-240-01-012524 | L40956-29 | PFOS |

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89591 (WG88961)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 3/18/24 16:40 | ADONA | 153% | 70-130% | ADONA and NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 3/18/24 16:40 | NFDHA | 68.7% | 70-130% | |
| CCV 3/18/24 20:17 | ADONA | 143% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW20B-GW-90-100-01-012424 | L40956-31 | 13C4-PFBA | 5.11 | R |
| OW20J-1-GW-310-320-01-012424 | L40956-32 | 13C2-4:2 FTS | 164 | No Qualifier |
| OW20P-1-GW-150-160-01-012424 | L40956-33 | 13C2-4:2 FTS | 167 | No Qualifier |
| OW20S-1-GW-190-200-01-012424 | L40956-34 | 13C4-PFBA | 26.8 | J+ |
| OW20S-3-GW-165-175-01-012424 | L40956-35 | 13C2-4:2 FTS | 171 | No Qualifier |
| MW21A-GW-410-420-01-012524 | L40956-37 | 13C4-PFBA | 46.3 | J+ |
| MW22B-GW-290-300-01-012924 | L40956-39 | 13C4-PFBA | 25.5 | J+ |
| MW22C-GW-160-170-01-012524 | L40956-40 | 13C4-PFBA | 21.9 | J+ |
| MW23A-GW-422-432-01-012524 | L40956-41 | 13C4-PFBA | 45.5 | J+ |
| MW23B-GW-299-309-01-012524 | L40956-42 | 13C4-PFBA | 39.6 | J+ |
| MW23C-GW-151-161-01-012524 | L40956-43 | 13C4-PFBA | 24.1 | J+ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG88961-102 / WG88961-103 | NFDHA | 67.4/66.6 | UJ |
| | 6:2 FTS | 108/132 | No Qualifier |
| | ADONA | 147/154 | No Qualifier |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW20P-1-GW-150-160-01-012424 | L40956-33 | PFOS |
| OW20S-1-GW-190-200-01-012424 | L40956-34 | PFOS |
| OW20T-1-GW-98-108-01-012424 | L40956-36 | PFOS |
| OW20S-3-GW-165-175-01-012424 | L40956-35 | PFOS |
| MW23C-GW-151-161-01-012524 | L40956-43 | PFOS |
| PL41-GW-72-82-03-012224 | L40956-58 | PFHpS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89591 (WG88961) Report date (mm/dd/yyyy): 4/15/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/10/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | No qualification is required for non-detect results associated with high recoveries. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | Non-detect results associated with low recoveries qualified as UJ. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate:<br>PL41-GW-72-82-01-012224 / PL41-GW-72-82-02-012224 / PL41-GW-72-82-03-012224 (RPDs ok) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | *with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**One result (for one sample) was rejected (R) due to a severely low labeled anaog recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/10/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG89709 (WG88962)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 3/19/24 16:55 | ADONA | 149% | 70-130% | ADONA and NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 3/19/24 20:45 | ADONA | 146% | 70-130% | |
| CCV 3/22/24 16:22 | ADONA | 147% | 70-130% | |
| CCV 3/22/24 16:22 | NFDHA | 65.0% | 70-130% | |

**Blanks (Section 3):** Blanks noncompliances are summarized below. The concentration of the target analyte detected in the method blank exceeded one-tenth of the concentration reported in the associated samples. The associated sample results are biased high and qualified as estimated (J). Qualification was not considered necessary for sample results reported as non-detect.

| Method Blank ID | Analyte | Concentration | RL | Units |
|---|---|---|---|---|
| WG88962-101 | PFOSA | 0.583 | 0.400 | ng/l |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW25B-GW-260-270-01-012524 | L40956-45 | FtS 4:2 ion-13C2 | 158 | No Qualifier |
| MW26A-GW-380-390-01-012524 | L40956-46 | Perfluorobutanoic acid-13C4 | 44.4 | J+ |
| PZGD-GW-35-40-01-012324 | L40956-52 | Perfluorotetradecanoic acid-13C2 | 48.8 | UJ |
| SP42-GW-116-126-01-012224 | L40956-59 | FtS 4:2 ion-13C2 | 181 | No Qualifier |
| EP16-WAT-BULK-02-012424 | L40956-66 | Hexafluoropropylene oxide dimer acid-13C3 | 44.2 | UJ |
| EP16-WAT-BULK-02-012424 | L40956-66 | Perfluorododecanoic acid-13C2 | 44.2 | UJ |
| EP16-WAT-BULK-02-012424 | L40956-66 | Perfluorooctanoic acid-13C8 | 49.4 | J+ |
| EP16-WAT-BULK-02-012424 | L40956-66 | Perfluorobutanoic acid-13C4 | 37.6 | J+ |
| EP16-WAT-BULK-02-012424 | L40956-66 | Perfluoroheptanoic acid-13C4 | 49.8 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP16-WAT-BULK-02-012424 | L40956-66 | Perfluorotetradecanoic acid-13C2 | 34.4 | UJ |
| EP16-WAT-BULK-02-012424 | L40956-66 | N-ethylperfluoro-1-octanesulfonamide-D5 | 40.3 | UJ |
| EP16-WAT-BULK-02-012424 | L40956-66 | FtS 6:2 ion-13C2 | 49.8 | UJ |
| WL6-WAT-BULK-01-012424 | L40956-67 | Perfluorobutanoic acid-13C4 | 48.5 | J+ |
| WL6-WAT-BULK-01-012424 | L40956-67 | Perfluorotetradecanoic acid-13C2 | 49.9 | UJ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| BL5-WAT-BULK-03-012424 | PFBA | **134**/89.6 | J+ |
| | PFOS | **193/143** | J+ |
| | PFDoS | 77.2/**61.4** | UJ |
| | ADONA | **145/141** | No qualifier |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG88962-102 | ADONA | 142 | No qualifier |

**Field Triplicates (Section 6Bi):**

Results qualified as estimated (UJ/J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **BL5-WAT-BULK-01-012424 / BL5-WAT-BULK-02-012424 / BL5-WAT-BULK-03-012424:** | | | | |
| PFMPA | 0.809 U | 0.808 U | 12.4 | **> ± 2xRL** |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW25B-GW-260-270-01-012524 | L40956-45 | Perfluorooctane sulfonate |
| MW25B-GW-260-270-01-012524 | L40956-45 | Perfluorononane sulfonate |
| MW25B-GW-260-270-01-012524 | L40956-45 | N-MeFOSA |
| MW26A-GW-380-390-01-012524 | L40956-46 | Perfluorooctane sulfonate |
| MW26B-GW-249-259-01-012524 | L40956-47 | Perfluorooctane sulfonate |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW26C-GW-130-140-01-012524 | L40956-48 | Perfluorooctane sulfonate |
| PZH-1-GW-90-100-01-012424 | L40956-54 | Perfluorooctane sulfonate |
| SP42-GW-116-126-01-012224 | L40956-59 | Perfluorodecane sulfonate |
| W6201-GW-109-124-01-012424 | L40956-61 | Perfluorooctane sulfonate |
| W6201-GW-109-124-01-012424 | L40956-61 | N-MeFOSA |
| W6201-GW-109-124-01-012424 | L40956-61 | Perfluorooctane sulfonate |
| W6201-GW-109-124-01-012424 | L40956-61 | N-MeFOSA |
| BL5-WAT-BULK-01-012424 | L40956-62 | N-MeFOSA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG89709 (WG88962) Report date (mm/dd/yyyy): 4/23/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/10/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | Detects were more than the RL:<br>PFOSA 0.583 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>EP16-WAT-BULK-01-012424 / EP16-WAT-BULK-02-012424<br>Field Triplicate:<br>BL5-WAT-BULK-01-012424 / BL5-WAT-BULK-02-012424 / BL5-WAT-BULK-03-012424 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>MSD RPDs ok<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/10/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90350 (WG89880)**

**Chain of custody preservation, and holding time (Section 1C):** Sample J-GW-72-76-01-042424 (L41282-15) was received at a temperature of 8.3°C, and above 6°C. Results were acceptable as the receipt temperature was <10°C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. The samples for this SDG were analyzed within 90 days.

**Estimated Maximum Possible Concentration (EMPC):**

PFOS results for samples Q3-GW-110-126-01-042224 (L41282-9), C-WT-GW-48-58-01-042424 (L41282-13), J-GW-72-76-01-042424 (L41282-15), C-OP-GW-134-143-01-042324 (L41282-21), and K-OP-GW-120-140-01-042324 (L41282-22) were reported as EMPCs. These results were qualified as estimated (J).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 05/24/24 20:51 | NFDHA | 271% | 70-130% | NFDHA results were not detected. No qualifiers. |
| CCV 05/25/24 00:30 | NFDHA | 252% | 70-130% | |
| CCV 05/25/24 03:16 | NFDHA | 271% | 70-130% | |
| CCV 06/5/24 16:33 | NFDHA | 41.6% | 70-130% | NFDHA results were nondetect and qualified UJ. |
| CCV 06/5/24 19:13 | NFDHA | 38.5% | 70-130% | |

**Blanks (Section 3Aii):** PFOSA was detected in Trip-Cooler1-042424 at a concentration of 0.476 J ng/L. The PFOSA result for samples L-GW-68-72-01-042424 (L41282-10) and C-WT-GW-48-58-01-042424 (L41282-13) were associated with this trip blank and were within 10 times the trip blank concentration. These results were qualified BJ.

**Labeled Analogs - surrogates (Section 4Bii):** Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Q3-GW-110-126-01-042224 | L41282-9 | 13C2-PFTeDA | 27.6 | UJ<br>PFTrDA: UJ |
| Q3-GW-110-126-01-042224 | L41282-9 | D5-N-EtFOSA | 43.6 | UJ |
| Q3-GW-110-126-01-042224 | L41282-9 | d9-NEt-FOSE | 44.9 | UJ |

**Laboratory Control Sample (Section 5Bi):** LCS noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS WG89880-102 | 6:2 FTS | 211% | 70-130% | NFDHA results were not detected. No qualifiers. |
| LCS WG89880-102 | NFDHA | 254% | 70-130% | |
| LCS WG89880-103 | NFDHA | 260% | 70-130% | |
| LCS WG89880-103 | 11Cl-PF3OUdS | 69.3% | 70-130% | 11Cl-PF3OUdS results were nondetect and qualified UJ. |

**Matrix Spike (Section 6Av):** Results associated with low recoveries were qualified and estimated as (UJ).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89880-104(MS)/105 (MSD) | PFBA* | **66.1**/114 | 70-130% | No qualifiers |
| | PFDoS | **68.6**/78.6 | 70-130% | UJ |
| | 11Cl-PF3OUdS | **63.3/68.5** | 70-130% | UJ |
| | NFDHA | **274/277** | 70-130% | No qualifiers (parent results were nondetect) |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Field Triplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Field Triplicate Result (ng/L) | RPD (%)/absolute difference |
|---|---|---|---|---|
| **L-GW-68-72-01-042424/L-GW-68-72-02-042424/L-GW-68-72-03-042424:** | | | | |
| PFHpA | 2.53 | 3.58 | 2.59 | 36.2 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 4095

Work order number/Lab report ID: DPWG90350 (WG89880) Report date (mm/dd/yyyy): 6/25/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/12/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | One sample was received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | Samples were analyzed within the SGS AXYS extended-time storage time limit of 90 days. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Yes, receipt temperature exceedances discussed. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-Cooler1-042324<br>Trip-Cooler2-042324<br>Trip-Cooler3-042324<br>Trip-Cooler1-042424 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Yes, see data qualifications summary for details and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample L-GW-68-72-03-042424. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | Low Level LCS |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>Q1-GW-60-64-01-042224/Q1-GW-60-64-02-042224<br>Field Triplicate: |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | L-GW-68-72-01-042424/L-GW-68-72-02-042424/L-GW-68-72-03-042424 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± RL, used for water matrix.<br>See data qualifications summary (attachment) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Andrei Niculau Title: Chemist III

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/12/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90213 (WG89881)**

**Chain of custody preservation, and holding time (Section 1C):** Sample EE-GW-74-84-01-042424 (L41282-16), EE-GW-74-84-02-042424 (L41282-17), and EE-GW-74-84-03-042424 (L41282-18) were received at a temperature of 8.3°C, and above 6°C. Results were acceptable as the receipt temperature was <10°C.

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. The samples for this SDG were analyzed within 90 days.

**Calibration (Section 2):** Calibration noncompliance are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 05/30/24 17:57 | NFDHA | 229% | 70-130% | NFDHA results were not detected. No qualifiers. |
| CCV 05/30/24 21:10 | NFDHA | 228% | 70-130% | |
| CCV 05/30/24 23:18 | NFDHA | 240% | 70-130% | |
| CCV 05/31/24 1:26 | NFDHA | 228% | 70-130% | |
| CCV 06/4/24 21:10 | NFDHA | 34.1% | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 06/4/24 22:30 | NFDHA | 41.0% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EQ-117189-043024 | L41305-4 | D5-EtFOSAA | 169 | No Qualifier |
| LEPR_1-GW-100-140-01-042524 | L41305-6 | 13C2-PFTeDA | 44.5 | UJ<br>PFTrDA: UJ |

**Laboratory Control Sample (Section 5Bi):** LCS noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS WG89881-103 | 6:2 FTS | 136% | 70-130% | Detects were qualified as estimated biased high (J+). |
| LCS WG89881-102 | 5:3 FTCA | 132% | 70-130% | Associated results were not detected. No qualifiers. |
| LCS WG89881-103 | 5:3 FTCA | 132% | 70-130% | |

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS WG89881-102 | PFEESA | 133% | 70-130% | |
| LCS WG89881-103 | PFEESA | 134% | 70-130% | |
| LCS WG89881-103 | MeFOSAA | 133% | 70-130% | |
| LCS WG89881-102 | NFDHA | 233% | 70-130% | |
| LCS WG89881-103 | NFDHA | 259% | 70-130% | |

**Matrix Spike (Section 6Av):** Recoveries for the MS for D3-N-MeFOSA and D5-N-EtFOSA were below 10% (LCS was ok). Sample results were qualified based the sample specific labeled analog recoveries as they are more representative.

Recoveries for the MS and MSD for PFBA, PFOA and NFDHA were above 150%.The PFBA and PFOA concentrations measured in the parent sample EE-GW-74-84-03-042424 (L41282-18) were greater than 4 times the amount spiked into the MS and MSD samples and results were not qualified. Recoveries above criteria for 5:3 FTCA, NFDHA, and PFEESA did not require qualification since these compounds were non detected in the associated sample.

**Estimated Maximum Possible Concentration (EMPC):**

PFOS results for samples R2-GW-100-104-01-042324 (L41282-24), LEPR_1-GW-100-140-01-042524 (L41305-6), DD-GW-63-73-01-042524 (L41305-7), HWY5_OP-GW-UNK-135-01-042524 (L41305-8), and BB3-GW-103-118-01-042524 (L41305-9) were reported as EMPCs. These results were qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 4095

Work order number/Lab report ID: DPWG90213 (WG89881) Report date (mm/dd/yyyy): 6/12/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/10/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Three samples were received at elevated temperatures. See data qualifications summary for details regarding receipt temperatures and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | Samples were analyzed within the SGS AXYS extended-time storage time limit of 90 days. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Yes, receipt temperature exceedances discussed |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-Cooler1-042524<br>Trip-Cooler2-042924<br>FieldBlank-043024<br>EQ-117189-043024<br>EQ-U104370X-043024 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | EQ-U104370X-043024:<br>PFBA 8.89 ng/L<br>PFOA 0.415 J ng/L<br>4/30/24 data not reported in this SDG. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample EE-GW-74-84-03-042424. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | Low Level LCS |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate:<br>EE-GW-74-84-01-042424/EE-GW-74-84-02- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 042424/EE-GW-74-84-03-042424 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Andrei Niculau Title: Chemist III

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/11/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90305 (WG89882)**

**Chain of custody, preservation and holding time (Section 1.E):**

SGS AXYS has completed an extended-time storage study and has determined that samples are stable for 90 days from sample collection when maintained at a temperature of -20 °C. The samples for this SDG were analyzed within 90 days.

**Calibration (Section 2):** Calibration noncompliance's are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/04/24 11:42 | ADONA | 168 | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 6/04/24 11:42 | NFDHA | 26.2 | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| CCV 6/04/24 16:00 | NFDHA | 40.4 | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| CCV 6/04/24 16:00 | ADONA | 169 | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 6/04/24 19:23 | NFDHA | 36.5 | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 6/04/24 19:23 | ADONA | 170 | 70-130% | ADONA results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 16:55 | 9Cl-PF3ONS | 136 | 70-130% | 9Cl-PF3ONS results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 16:55 | 11Cl-PF3OUdS | 131 | 70-130% | 11Cl-PF3OUdS results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 16:55 | 7:3 FTCA | 61.2 | 70-130% | 7:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 16:55 | NFDHA | 34.8 | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 16:55 | ADONA | 186 | 70-130% | ADONA results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 19:24 | ADONA | 193 | 70-130% | ADONA results were not reported from these CCVs. No qualifiers. |
| CCV 6/07/24 19:24 | 9Cl-PF3ONS | 143 | 70-130% | 9Cl-PF3ONS results were not reported from these CCVs. No qualifiers |
| CCV 6/07/24 19:24 | 11Cl-PF3OUdS | 133 | 70-130% | 11Cl-PF3OUdS results were not reported from these CCVs. No qualifiers |
| CCV 6/07/24 19:24 | 7:3 FTCA | 60.2 | 70-130% | 7:3 FTCA results were not reported from these CCVs. No qualifiers. |

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 7/04/24 19:24 | NFDHA | 34.1 | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |

**Blanks (Section 3Aii):**

The analyte 6:2 FTS (120 ng/L) was detected in Trip-cooler2-050624. Sample results did not require qualification based on the associated blank if the sample result was reported as not detected or was detected at a concentration above the laboratory reporting limit and significantly higher than the associated blank concentration. The analyte PFOSA (0.565 ng/L) was detected in the sample Field blank-050624. Data were qualified as detected and flagged as 'BJ' if the associated sample result was reported between the laboratory reporting limit (RL) and blank action level.

**Laboratory control spike/Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ) for the non detects. Results associated with high recoveries were nondetects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89882-102/103 | ADONA | **167/172** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89882-102/103 | NFDHA | **35.7/43.5** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Q2-GW-80-84-01-042924 | L41305-14 | 13C2-PFTeDA | 47.2 | UJ PFTrDA: UJ |
| I-GW-54-59-01-043024 | L41305-16 | 13C2-PFTeDA | 47.9 | UJ PFTrDA: UJ |
| R-WT-GW-50-60-01-043024 | L41305-18 | 13C2-PFTeDA | 43.4 | UJ PFTrDA: UJ |
| EQ-GEOSUB-050624 | L41329-4 | 13C2-PFTeDA | 48.8 | UJ PFTrDA: UJ |
| PL41-GW-72-82-01-050624 | L41329-6 | 13C2-PFTeDA | 48.8 | UJ PFTrDA: UJ |
| PL41-GW-72-82-03-050624 | L41329-8 (A) | 13C2-PFTeDA | 42.7 | UJ PFTrDA: UJ |
| OD3-WAT-BULK-01-050624 | L41329-10 | 13C2-PFTeDA | 41.4 | UJ PFTrDA: UJ |
| OD5-WAT-BULK-01-050624 | L41329-12 | 13C2-PFTeDA | 45.0 | UJ PFTrDA: UJ |

**Matrix spike/ Matrix spike duplicates – (Section 6AV).** Results associated with low recoveries were qualified and estimated as (UJ).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89882-104(MS)/105 (MSD) | **PFOA*** | **34.6/40.5** | 70-130% | No qualifiers |
| | **PFDoS** | **68.1/**72.0 | 70-130% | UJ |
| | **ADONA** | **165/169** | 70-130% | No qualifiers (parent result was nondetect) |
| | **NFDHA** | **38.1/44.7** | 70-130% | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Field Triplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | Field Triplicate Result (ng/g) | RPD (%)/absolute difference |
|---|---|---|---|---|
| **PL41-GW-72-82-01-050624/PL41-GW-72-82-02-050624/PL41-GW-72-82-03-050624:** | | | | |
| PFOSA | 0.594 J | 2.35 | 1.8 | >±RL |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90305 (WG89882) Report date (mm/dd/yyyy): 6/17/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/11/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | Samples were analyzed within the SGS AXYS extended-time storage study time limit of 90 days. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip-cooler1-050624<br>Trip-cooler2-050624<br>Field blank-050624<br>EQ-GEOSUB-050624<br>EQ-Dipper-050624 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects were more than the RL<br>6:2 FTS 120 ng/L in Trip-cooler2-050624<br>PFOSA 0.565 J ng/L in Field blank-050624 |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample PL41-GW-72-82-03-050624 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate:<br>PL41-GW-72-82-01-050624 / PL41-GW-72- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 82-02-050624 / PL41-GW-72-82-03-050624 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± RL, used for water matrix.<br>MS/MSD RPDs ok.<br>Field triplicate: See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/11/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90198 (WG89883)**

**Calibration (Section 2): Calibration** noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 5/25/24 20:02 | 3:3 FTCA | **135%** | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 5/25/24 20:02 | NFDHA | **273%** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 5/25/24 23:40 | NFDHA | **241%** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 5/25/24 02:27 | NFDHA | **242%** | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |
| CCV 5/25/24 05:14 | NFDHA | **241%** | 70-130% | NFDHA results were not reported from these CCVs. No qualifiers. |

**Laboratory control spike/ Laboratory control spike duplicates – (Section 5Bii):** LSC/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89883-102/103 | 3:3 FTCA | 130/**145** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89883-102/103 | NFDHA | **235/321** | 70-130% | All the associated sample results were not detected. No qualifiers |
| WG89883-102/103 | 5:3 FTCA | 127/**148** | 70-130% | All the associated sample results were not detected. No qualifiers |
| WG89883-102/103 | PFEESA | 121/**141** | 70-130% | All the associated sample results were not detected. No qualifiers |

**Matrix spike/ Matrix spike duplicates – (Section 6AV).** Results associated with high recoveries were nondetects and were acceptable without qualification. Results associated with low recoveries were qualified as estimated as (UJ).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89883-104 (MS)/105(MSD) | **PFOA*** | **4.01/NA** | 70-130% | No qualifiers |
| WG89883-104 (MS)/105(MSD) | **PFOS**** | **192**/124 | 70-130% | No qualifiers |
| WG89883-104 (MS)/105(MSD) | **5:3 FTCA** | **133/132** | 70-130% | No qualifiers |

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89883-104 (MS)/105(MSD) | **PFEESA** | **132/133** | 70-130% | No qualifiers |
| WG89883-104 (MS)/105(MSD) | **NFDHA** | **237/289** | 70-130% | No qualifiers |
| WG89883-105 (MSD) | **8:2 FTS** | 79.9/**68.8** | 70-130% | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines.).

** Samples RC5-WAT-BULK-03-050624(5X) was reanalyzed due to calibration exceedances required dilutions. These samples were diluted beyond the laboratory's ability to quantitate MS/MSD recoveries. As such, no further action was required, and data qualification was not considered necessary.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90198 (WG89883) Report date (mm/dd/yyyy): 6/12/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/11/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample RC5-WAT-BULK-03-050624 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate:<br>RC5-WAT-BULK-01-050624/RC5-WAT-BULK-02-050624/RC5-WAT-BULK-03-050624 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>MS/MSD RPDs ok (not all compounds reported). Field triplicate RPDs ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/11/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90423 (WG89934)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/06/24 02:23 | ADONA | 178% | 70-130% | ADONA results were nondetect (no qualifiers). Associated 7:3 FTCA, NFDHA, and 9Cl-PF3ONS results were nondetect and qualified as estimated (UJ). |
| | 7:3 FTCA | 60.3% | 70-130% | |
| | NFDHA | 25.6% | 70-130% | |
| CCV 9/06/24 06:27 | ADONA | 166% | 70-130% | |
| | 7:3 FTCA | 58.1% | 70-130% | |
| | NFDHA | 43.1% | 70-130% | |
| CCV 9/06/24 09:36 | ADONA | 175% | 70-130% | |
| | 7:3 FTCA | 60.0% | 70-130% | |
| | NFDHA | 31.7% | 70-130% | |
| CCV 9/06/24 12:16 | ADONA | 187% | 70-130% | |
| | 9Cl-PF3ONS | 143% | 70-130% | |
| | 7:3 FTCA | 60.7% | 70-130% | |
| | NFDHA | 40.8% | 70-130% | |

**Blanks (Section 3):** Blanks noncompliances are summarized below. The concentration of the target analyte detected in the Trip blank exceeded the RL. Detects within 10 times the blank concentration were qualified B.

| Trip Blank ID | Analyte | Concentration | RL | Units |
|---|---|---|---|---|
| Trip-cooler2-050724 | PFOS | 3.39 | 0.402 | ng/l |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Detects associated with high recoveries were qualified J-. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Trip-cooler1-050724 | L41344-1 | 13C3-HFPO-DA | 45.7 | UJ |
| Trip-cooler2-050724 | L41344-2 | 13C3-HFPO-DA | 47.3 | UJ |
| RC23-WAT-BULK-01-050624 | L41344-3 | 13C3-HFPO-DA | 49.5 | UJ |
| EP26D-WAT-BULK-01-050724 | L41344-4 | 13C2-4:2 FTS | 156 | No qualifier |
| EP26D-WAT-BULK-01-050724 | L41344-4 | 13C3-HFPO-DA | 46.1 | UJ |
| EP16-WAT-BULK-01-050724 | L41344-5 | 13C2-PFTeDA | 42.9 | UJ<br>PFTrDA: UJ |
| EP16-WAT-BULK-01-050724 | L41344-5 | 13C8-PFOSA | 142 | No qualifier |
| EP16-WAT-BULK-01-050724 | L41344-5 | 13C3-HFPO-DA | 48.1 | UJ |
| WL6-WAT-BULK-01-050724 | L41344-6 | 13C2-PFTeDA | 45.6 | UJ<br>PFTrDA: UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL6-WAT-BULK-01-050724 | L41344-6 | 13C3-HFPO-DA | 48.3 | UJ |
| WL15-WAT-BULK-01-050724 | L41344-7 | 13C2-PFTeDA | 36.6 | UJ<br>PFTrDA: UJ |
| WL15-WAT-BULK-01-050724 | L41344-7 | 13C3-HFPO-DA | 46.3 | UJ |
| WC2-WAT-BULK-01-050724 | L41344-8 | 13C4-PFBA | 31.6 | J+ |
| WC2-WAT-BULK-01-050724 | L41344-8 | 13C2-PFTeDA | 40.4 | UJ<br>PFTrDA: UJ |
| WC2-WAT-BULK-01-050724 | L41344-8 | 13C3-HFPO-DA | 49.5 | UJ |
| WC1-WAT-BULK-01-050724 | L41344-9 | 13C2-PFTeDA | 46.1 | UJ<br>PFTrDA: UJ |
| MW2B-GW-57-62-01-050724 | L41344-10 | 13C3-HFPO-DA | 48.7 | UJ |
| MW1C-GW-40-45-01-050724 | L41344-11 | 13C2-PFTeDA | 47.1 | UJ<br>PFTrDA: UJ |
| MW14B-GW-60-70-01-050724 | L41344-12 | 13C2-PFTeDA | 47.2 | UJ<br>PFTrDA: UJ |
| MW14B-GW-60-70-01-050724 | L41344-13 | 13C3-HFPO-DA | 47.7 | UJ |
| MW14B-GW-60-70-02-050724 | L41344-14 | 13C8-PFOSA | 168 | J- |
| MW14B-GW-60-70-02-050724 | L41344-14 | 13C3-HFPO-DA | 47.2 | UJ |
| MW2F-GW-130-150-01-050724 | L41344-15 | 13C3-HFPO-DA | 45.6 | UJ |
| MW2F-GW-130-150-02-050724 | L41344-16 | 13C3-HFPO-DA | 49.7 | UJ |
| MW2F-GW-130-150-03-050724 | L41344-17 | 13C8-PFOSA | 154 | No qualifier |
| MW2F-GW-130-150-03-050724 | L41344-17 | 13C3-HFPO-DA | 48.2 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG89934-102/ WG89934-103 | 6:2 FTS | 118/**136** | No qualifier |
| | ADONA | **173/172** | No qualifier |
| | 7:3 FTCA | **59.2/61.5** | Associated results were nondetect and qualified UJ. |
| | NFDHA | **42.3/28.3** | |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| MW2F-GW-130-150-03-050724 | PFBA* | **67.8/65.1** | No qualifier |
| | PFOA* | **/27.6** | No qualifier |
| | ADONA | **173/167** | No qualifier |
| | 9Cl-PF3ONS | **131**/121 | No qualifier |
| | 7:3 FTCA | **61.8/60.7** | UJ |
| | NFDHA | **35.3/35.2** | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WL6-WAT-BULK-01-050724 | L41344-6 | PFHpS |
| MW14B-GW-60-70-01-050724 | L41344-13 | MeFOSAA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90423 (WG89934) Report date (mm/dd/yyyy): 6/25/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/11/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-cooler1-050724<br>Trip-cooler2-050724 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects were more than the RL:<br>PFOS 3.39 ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW2F-GW-130-150-03-050724 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>MW1C-GW-40-45-01-050724/MW1C-GW-40-45-02-050724<br>Field Triplicate:<br>MW2F-GW-130-150-01-05072/MW2F-GW-130-150-02-050724/MW2F-GW-130-150-03-050724 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/11/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90380 (WG89935)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/04/24 22:30 | ADONA | 172% | 70-130% | ADONA results were not nondetect and were acceptable without qualification. NFDHA results were qualified UJ. |
| CCV 6/04/24 22:30 | NFDHA | 41.0% | 70-130% | |
| CCV 6/05/24 02:05 | ADONA | 163% | 70-130% | |
| CCV 6/05/24 02:05 | NFDHA | 43.4% | 70-130% | |
| CCV 6/05/24 05:42 | ADONA | 162% | 70-130% | |
| CCV 6/05/24 05:42 | NFDHA | 36.8% | 70-130% | |

**Blanks (Section 3):** Blank noncompliances are summarized below. Qualification was not considered necessary for sample results reported as non-detect. Detects within 10 times the blank concentration were qualified B/BJ.

| Trip Blank ID | Analyte | Concentration | RL | Units |
|---|---|---|---|---|
| Trip-cooler2-050824 | PFHxA | 1.94 | 0.400 | ng/l |
| | PFHpA | 0.487 J | 0.400 | ng/l |
| | N-MeFOSA | 0.418 J | 0.400 | ng/l |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Trip-cooler3-050824 | L41350-3 | 13C2-PFTeDA | 47.8 | UJ PFTrDA: UJ |
| EQ-Peritubing-050824 | L41350-4 | 13C2-PFTeDA | 46.1 | UJ PFTrDA: UJ |
| EQ-Bailer-050824 | L41350-5 | 13C2-PFTeDA | 43.2 | UJ PFTrDA: UJ |
| OW7Q-1-GW-64-74-01-050824 | L41350-6 | 13C2-PFTeDA | 39.1 | UJ PFTrDA: UJ |
| OW7Q-1-GW-64-74-02-050824 | L41350-7 | 13C2-PFTeDA | 38.9 | UJ PFTrDA: UJ |
| OW7Q-1-GW-64-74-03-050824 | L41350-8 (A) | 13C2-PFTeDA | 40.0 | UJ PFTrDA: UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZH-2-GW-40-50-01-050824 | L41350-12 | 13C2-PFTeDA | 39.8 | UJ<br>PFTrDA: UJ |
| MW15A-GW-330-340-01-050824 | L41350-13 | 13C4-PFBA | 16.6 | UJ |
| MW15A-GW-330-340-01-050824 | L41350-13 | 13C2-PFTeDA | 46.1 | UJ<br>PFTrDA: UJ |
| MW15A-GW-330-340-01-050824 | L41350-13 | 13C2-4:2 FTS | 177 | No Qualifier |
| MW12A-GW-350-360-01-050824 | L41350-14 | 13C4-PFBA | 13.0 | J+ |
| MW12A-GW-350-360-01-050824 | L41350-14 | 13C2-PFTeDA | 46.0 | UJ<br>PFTrDA: UJ |
| MW9B-GW-90-100-01-050824 | L41350-15 | 13C4-PFBA | 11.6 | J+ |
| MW9B-GW-90-100-01-050824 | L41350-15 | 13C2-4:2 FTS | 170 | No Qualifier |
| MW9A-GW-140-150-01-050824 | L41350-16 | 13C4-PFBA | 10.3 | J+ |
| OW7S-1-GW-90-100-01-050824 | L41350-18 | 13C2-4:2 FTS | 182 | No Qualifier |
| MW7A-GW-200-210-01-050824 | L41350-19 | 13C4-PFBA | 9.97 | J+ |
| MW7A-GW-200-210-01-050824 | L41350-19 | 13C2-4:2 FTS | 166 | No Qualifier |
| OW7S-3-GW-100-110-01-050824 | L41350-20 | 13C4-PFBA | 40.4 | J+ |
| MW6A-GW-185-192-01-050824 | L41350-21 | 13C4-PFBA | 7.03 | R |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG89935-102/ WG89935-103 | PFDoS | 70.9/**67.1** | UJ |
| | ADONA | **166/169** | No Qualifier |
| | NFDHA | **39.4/40.5** | UJ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| OW7Q-1-GW-64-74-03-050824 | PFDoS | 71.0/**66.0** | UJ |
| | ADONA | **154/150** | No Qualifier |
| | NFDHA | **43.0/46.8** | UJ |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| Trip-cooler2-050824 | L41350-2 | N-MeFOSA |
| MW6A-GW-185-192-01-050824 | L41350-21 | PFOS |
| MW7A-GW-200-210-01-050824 | L41350-19 | PFOS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW15A-GW-330-340-01-050824 | L41350-13 | PFOS |
| MW12A-GW-350-360-01-050824 | L41350-14 | PFOS |
| MW8A-GW-202-212-01-050824 | L41350-17 | PFNA |
| OW7S-1-GW-90-100-01-050824 | L41350-18 | PFNA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90380 (WG89935) Report date (mm/dd/yyyy): 6/25/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/12/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Trip-cooler3-050824, EQPeritubing-050824 and EQ-Bailer-050824 samples were received above the recommended temperature limit (>6°C). Results were not qualified as the receipt temperature was 6.5°C, and was <10°C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-cooler1-050824<br>Trip-cooler2-050824<br>Trip-cooler3-050824<br>EQ-Peritubing-050824<br>EQ-Bailer-050824 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects were above than the RL:<br>PFHxA 1.94 ng/l<br>PFHpA 0.487 ng/l<br>N-MeFOSA 0.418 ng/l |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | OW7Q-1-GW-64-74-03-050824 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | ☒ | ☐ | ☐ | MSD RPDs<br>Field Triplicate:<br>OW7Q-1-GW-64-74-01-050824/OW7Q-1-GW-64-74-02-050824/OW7Q-1-GW-64-74-03-050824 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>MS/MSD RPDs ok. Field triplicate RPDs ok. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications. One result was rejected (R) due to a severely low labled analog (EIS) recovery.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/12/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90389 (WG89936)**

**Chain of custody, preservation and holding time (Section 1.G):**

The laboratory narrative noted that the sample Trip-Cooler2-042424 receipt temperature (8.3°C) did not meet the method requirement criteria (<6°C) for the analysis of PFAS. Results were not qualified as the receipt temperature was <10°C.

**Calibration (Section 2): Calibration** noncompliance's are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/07/24 19:24 | 9Cl-PF3ONS | **143%** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 6/07/24 19:24 | 11Cl-PF3OUdS | **132%** | 70-130% | |
| CCV 6/07/24 19:24 | 7:3 FTCA | **60.2%** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| CCV 6/07/24 19:24 | NFDHA | **34.1%** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| CCV 6/07/24 19:24 | ADONA | **193%** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 6/07/24 22:47 | ADONA | **175%** | 70-130% | Post analysis CCV. Results qualified as listed above (exceedances are similar). |
| CCV 6/07/24 22:47 | 9Cl-PF3ONS | **132%** | 70-130% | |
| CCV 6/07/24 22:47 | 7:3 FTCA | **58.6** | 70-130% | |
| CCV 6/07/24 22:47 | NFDHA | **41.1** | 70-130% | |

**Laboratory control spike/ Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ). Results associated with high recoveries were nondetects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89936-102/103 | ADONA | **191/199** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89936-102/103 | NFDHA | **34.7/33.5** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG89936-102/103 | PFHpA | 109 **/137** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89936-102/103 | 6:2 FTS | 121**/153** | 70-130% | All the associated sample results were not detected. No qualifiers. |

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG89936-102/103 | 7:3 FTCA | **62.9**/**62.9** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG89936-102/103 | EtFOSAA | 99.9/**134** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89936-102/103 | 9Cl-PF3ONS | **144/145** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89936-102/103 | 11Cl-PF3OUdS | 125/**133** | 70-130% | All the associated sample results were not detected. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Trip-Cooler2-042424 | L41282-5 R | 13C2-PFTeDA | 41.6 | UJ<br>PFTrDA: UJ |
| Trip-Cooler2-042424 | L41282-5 R | 13C3-HFPO-DA | 48.3 | UJ |
| M-OP-GW-127-167-01-042224 | L41282-6 R | 13C2-PFTeDA | 41.4 | UJ<br>PFTrDA: UJ |
| M-OP-GW-127-167-01-042224 | L41282-6 R | 13C3-HFPO-DA | 46.1 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| M-OP-GW-127-167-01-042224 | L41282-6 R | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90389 (WG89936) Report date (mm/dd/yyyy): 6/20/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | The receipt temperature (8.3°C) for sample Trip-Cooler2-042424 did not meet the requirement criteria (<6°C). |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-Cooler2-042424 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90418 (WG90103)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/12/24 02:09 | 9Cl-PF3ONS | 136% | 70-130% | 9Cl-PF3ONS and ADONA results were were nondetect and were acceptable without qualification.7:3 FTCA and NFDHA results were not detected and qualified UJ. |
| CCV 06/12/24 02:09 | 7:3 FTCA | 61.6% | 70-130% | |
| CCV 06/12/24 02:09 | NFDHA | 37.0% | 70-130% | |
| CCV 06/12/24 05:31 | ADONA | 169% | 70-130% | |
| CCV 06/12/24 05:31 | 7:3 FTCA | 62.4% | 70-130% | |
| CCV 06/12/24 05:31 | NFDHA | 29.8% | 70-130% | |
| CCV 06/12/24 07:32 | ADONA | 182% | 70-130% | |
| CCV 06/12/24 07:32 | 9Cl-PF3ONS | 138% | 70-130% | |
| CCV 06/12/24 07:32 | 7:3 FTCA | 59.3% | 70-130% | |
| CCV 06/12/24 07:32 | NFDHA | 28.5% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Three results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW8B-GW-50-60-02-050824 | L41350-10 | 3C2-PFTeDA | 48.1 | UJ<br>PFTrDA: UJ |
| MW8B-GW-50-60-03-050824 | L41350-11 (A) | 13C3-HFPO-DA | 49.7 | UJ |
| MW6B-GW-140-150-01-050824 | L41350-22 | 13C4-PFBA | 4.03 | R |
| MW6B-GW-140-150-01-050824 | L41350-22 | 13C2-4:2 FTS | 154 | No Qualifier |
| MW6B-GW-140-150-01-050824 | L41350-22 | D3-MeFOSAA | 159 | No Qualifier |
| MW6B-GW-140-150-01-050824 | L41350-22 | D5-EtFOSAA | 160 | No Qualifier |
| MW6B-GW-140-150-01-050824 | L41350-22 | 13C3-HFPO-DA | 45.4 | UJ |
| MW13B-GW-295-305-01-050824 | L41350-24 | 13C4-PFBA | 9.83 | R |
| MW13B-GW-295-305-01-050824 | L41350-24 | 13C2-4:2 FTS | 234 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13B-GW-295-305-01-050824 | L41350-24 | D3-MeFOSAA | 176 | No Qualifier |
| MW13B-GW-295-305-01-050824 | L41350-24 | D5-EtFOSAA | 181 | No Qualifier |
| MW13B-GW-295-305-01-050824 | L41350-24 | 13C3-HFPO-DA | 46.7 | UJ |
| MW13C-GW-115-125-01-050824 | L41350-25 | 13C2-PFTeDA | 44.6 | UJ<br>PFTrDA: UJ |
| MW13C-GW-115-125-01-050824 | L41350-25 | 13C3-HFPO-DA | 47.4 | UJ |
| MW3A-GW-230-250-01-050824 | L41350-26 | 13C2-PFTeDA | 49.2 | UJ<br>PFTrDA: UJ |
| MW3A-GW-230-250-01-050824 | L41350-26 | D3-MeFOSAA | 172 | No Qualifier |
| MW3A-GW-230-250-01-050824 | L41350-26 | D5-EtFOSAA | 180 | No Qualifier |
| MW3A-GW-230-250-01-050824 | L41350-26 | 13C3-HFPO-DA | 47.8 | UJ |
| MW3B-GW-110-130-01-050824 | L41350-27 | 13C4-PFBA | 4.84 | R |
| MW3B-GW-110-130-01-050824 | L41350-27 | 13C2-4:2 FTS | 221 | No Qualifier |
| MW3B-GW-110-130-01-050824 | L41350-27 | D3-MeFOSAA | 172 | No Qualifier |
| MW3B-GW-110-130-01-050824 | L41350-27 | D5-EtFOSAA | 177 | No Qualifier |
| MW3B-GW-110-130-01-050824 | L41350-27 | 13C3-HFPO-DA | 45.1 | UJ |
| EQ-HydraSleeve-051324 | L41350-7 | 13C2-PFTeDA | 44.2 | UJ<br>PFTrDA: UJ |
| EQ-HydraSleeve-051324 | L41350-7 | 13C3-HFPO-DA | 49.4 | UJ |
| W2D-GW-7-17-02-050924 | L41350-8 | 13C3-HFPO-DA | 47.8 | UJ |
| MW2D-GW-7-17-01-050924 | L41350-47 | 13C3-HFPO-DA | 47.0 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG90103-102 | 7:3 FTCA | 60.3 | UJ |
| | NFDHA | 34.8 | UJ |
| WG90103-103 | 6:2 FTS | 136 | No qualifier |
| | ADONA | 180 | No qualifier |
| | 9Cl-PF3ONS | 132 | No qualifier |
| | 7:3 FTCA | 64.4 | UJ |
| | NFDHA | 36.5 | UJ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| MW8B-GW-50- | PFBA* | 122/**142** | No qualifier |

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| 60-03-050824 | PFOA* | 121/**158** | No qualifier |
| | PFOS* | 126/**162** | No qualifier |
| | ADONA | **166/170** | No qualifier |
| | 9Cl-PF3ONS | 127/**132** | No qualifier |
| | 7:3 FTCA | **62.6/59.0** | UJ |
| | NFDHA | **39.7/38.9** | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW6B-GW-140-150-01-050824 | L41350-22 | PFOS |

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90418 (WG90103) Report date (mm/dd/yyyy): 6/25/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | PZH-3-GW-30-40-01-050824', 'MW13B-GW-295-305-01-050824', 'MW13C-GW-115-125-01-050824', 'MW3A-GW-230-250-01-050824' and 'MW3B-GW-110-130-01-050824 samples were received at 6.3 °C and above the recommended temperature limit (6°C). Results were not qualified as the receipt temperature was <10°C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip-cooler1-050924<br>Trip-cooler2-050924<br>Trip-cooler3-050924<br>Trip-cooler4-051024<br>Trip-cooler5-051324<br>Trip-cooler6-051324<br>EQ-HydraSleeve-051324 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | EQ-HydraSleeve-051324:<br>PFOSA 0.948 J ng/L (less than the RL)<br>05/13/2026 results not reported in this SDG. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>MW2D-GW-7-17-01-050924/MW2D-GW-7-17-02-050924<br>Field Triplicate:<br>MW8B-GW-50-60-01-050824/MW8B-GW-50-60-02-050824/MW8B-GW-50-60-03-050824 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications. Three results were rejected (R) due to severely low labeled analog (EIS) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/14/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90470 (WG90105)**

**Estimated Maximum Possible Concentration (EMPC):**

The following results were reported as an EMPC. These results were qualified as estimated (J).

| Sample | Analyte |
|---|---|
| MW2C-GW-35-40-01-050924 | N-MeFOSA |
| W6102-GW-93-105-01-050924 | PFDS |
| W6102-GW-93-105-01-050924 | N-MeFOSA |
| W6201-GW-109-124-01-050924 | N-MeFOSA |
| MW14D-GW-6-21-02-050924 | N-MeFOSA |
| MW17C-GW-38.5-48.5-01-050924 | N-MeFOSA |
| PZH-1-GW-90-100-01-050924 | PFOS |
| MW17B-GW-90-100-01-050924 | PFOS |
| MW14A-GW-316-326-01-050924 | N-MeFOSA |
| MW20A-GW-130-140-01-050924 | PFNS |
| OW20S-1-GW-190-200-01-050924 | PFOS |
| OW20S-1-GW-190-200-01-050924 | N-MeFOSA |
| OW20P-1-GW-150-160-01-050924 | PFOS |
| OW20T-1-GW-98-108-01-050924 | PFOS |
| OW20S-2-GW-200-210-01-050924 | PFOS |
| OW20S-2-GW-200-210-01-050924 | N-MeFOSA |
| OW20S-3-GW-165-175-01-050924 | PFOS |
| MW18A-GW-352-362-01-050924 | PFOS |
| MW22A-GW-429-439-01-050924 | N-MeFOSA |

N-MeFOSA in the above samples, were qualified BJ for laboratory blank detection.

**Calibration (Section 2):** Calibration noncompliance are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/19/24 13:37 | ADONA | 188% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 13:37 | 9Cl-PF3ONS | 135% | 70-130% | |
| CCV 06/19/24 13:37 | 11Cl-PF3OUdS | 145% | 70-130% | |
| CCV 06/19/24 17:52 | ADONA | 189% | 70-130% | Results were not detected. No qualifiers. |

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/19/24 17:52 | 9Cl-PF3ONS | 136% | 70-130% | |
| CCV 06/19/24 17:52 | 11Cl-PF3OUdS | 151% | 70-130% | |
| CCV 06/19/24 19:13 | ADONA | 193% | 70-130% | |
| CCV 06/19/24 19:13 | 9Cl-PF3ONS | 140% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 19:13 | 11Cl-PF3OUdS | 150% | 70-130% | |
| CCV 06/19/24 21:27 | ADONA | 183% | 70-130% | |
| CCV 06/19/24 21:27 | 9Cl-PF3ONS | 133% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 21:27 | 11Cl-PF3OUdS | 142% | 70-130% | |
| CCV 06/19/24 13:37 | NFDHA | 38.5% | 70-130% | |
| CCV 06/19/24 17:52 | NFDHA | 41.0% | 70-130% | All samples, UJ |
| CCV 06/19/24 19:13 | NFDHA | 37.1% | 70-130% | |
| CCV 06/19/24 21:27 | NFDHA | 40.8% | 70-130% | |

**Blanks (Section 3Bii):** The N-MeFOSA result for samples MW2C-GW-35-40-01-050924 (L41364-9), W6102-GW-93-105-01-050924 (L41364-10), W6201-GW-109-124-01-050924 (L41364-11), MW14D-GW-6-21-02-050924 (L41364-13), MW17C-GW-38.5-48.5-01-050924 (L41364-14), MW14A-GW-316-326-01-050924 (L41364-17), OW20S-1-GW-190-200-01-050924 (L41364-19), OW20S-2-GW-200-210-01-050924 (L41364-22), and MW22A-GW-429-439-01-050924 (L41364-26) were within 10 times the trip blank concentration and qualified BJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+) and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Seven results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| W6201-GW-109-124-01-050924 | L41364-11 | 13C2-PFTeDA | 41.5 | UJ<br>PFTrDA: UJ |
| PZH-1-GW-90-100-01-050924 | L41364-15 | d7-NMe-FOSE | 164 | None |
| PZH-1-GW-90-100-01-050924 | L41364-15 | d9-NEt-FOSE | 181 | None |
| MW14A-GW-316-326-01-050924 | L41364-17 | 13C4-PFBA | 8.33 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW14A-GW-316-326-01-050924 | L41364-17 | 13C2-4:2 FTS | 169 | None |
| MW20A-GW-130-140-01-050924 | L41364-18 | 13C4-PFBA | 6.5 | R |
| OW20S-1-GW-190-200-01-050924 | L41364-19 | 13C4-PFBA | 21.4 | J+ |
| OW20S-1-GW-190-200-01-050924 | L41364-19 | 13C2-8:2 FTS | 38.3 | UJ |
| OW20S-1-GW-190-200-01-050924 | L41364-19 | 13C8-PFOSA | 179 | None |
| OW20S-1-GW-190-200-01-050924 | L41364-19 | d7-NMe-FOSE | 160 | None |
| OW20P-1-GW-150-160-01-050924 | L41364-20 | 13C4-PFBA | 5.38 | R |
| OW20T-1-GW-98-108-01-050924 | L41364-21 | 13C4-PFBA | 5.31 | R |
| OW20T-1-GW-98-108-01-050924 | L41364-21 | 13C3-HFPO-DA | 49.8 | UJ |
| OW20S-2-GW-200-210-01-050924 | L41364-22 | 13C4-PFBA | 3.08 | R |
| OW20S-2-GW-200-210-01-050924 | L41364-22 | 13C5-PFPeA | 48.2 | J+ |
| OW20J-2-GW-310-320-01-050924 | L41364-23 | 13C4-PFBA | 6.85 | R |
| OW20J-2-GW-310-320-01-050924 | L41364-23 | 13C3-HFPO-DA | 48.5 | UJ |
| OW20S-3-GW-165-175-01- 050924 | L41364-24 | 13C4-PFBA | 10.1 | J+ |
| OW20S-3-GW-165-175-01- 050924 | L41364-24 | 13C2-4:2 FTS | 164 | None |
| MW18A-GW-352-362-01-050924 | L41364-25 | 13C4-PFBA | 9.54 | R |
| MW22A-GW-429-439-01-050924 | L41364-26 | 13C4-PFBA | 19.0 | J+ |
| MW22B-GW-290-300-01-050924 | L41364-27 | 13C4-PFBA | 24.4 | J+ |

**Laboratory Control Sample (Section 5Bi):** Recoveries for the LCS are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS/LCSD WG90105-102 WG90105-104 | ADONA | 173%/177% | 70-130% | ADONA was not detected, no qualification. |
| LCS/LCSD WG90105-102 WG90105-104 WG90105-103 | NFDHA | 39%/44%/ 50.4 | 70-130% | UJ all samples. |
| LCS WG90105-103 | ADONA | 182% | 70-130% | ADONA was not detected, no qualification. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 4095

Work order number/Lab report ID: DPWG90470 (WG90105) Report date (mm/dd/yyyy): 6/27/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/12/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | Yes, see data qualifications summary for details and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☐ | ☐ | ☒ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☐ | ☒ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD<br>Field Duplicate:<br>MW14D-GW-6-21-01-050924/MW14D-GW-6-21-02-050924 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | PZH-1-GW-90-100-01-050924 (L41364-15) and MW17B-GW-90-100-01-050924 (L41364-16) had elevated limits as not all the sample passed through the SPE column during sample prep due to matrix. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis.**

**See attachment for additional comments and qualifications. Seven results were rejected (R) due to severely low labeled analog (EIS) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Andrei Niculau Title: Chemist III

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/12/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90486 (WG90106)**

**Estimated Maximum Possible Concentration (EMPC):**

The following results were reported as an EMPC. These results were qualified as estimated (J).

| Sample | Analyte |
|---|---|
| MW21A-GW-410-420-01-051324 | PFNA |
| MW10A-GW-273-283-01-051324 | PFPeS |
| MW18B-GW-225-235-01-050924 | PFOS |
| MW13A-GW-350-370-01-051024 | PFOS |
| MW17A-GW-230-240-01-051324 | PFOS |
| MW10A-GW-273-283-01-051324 | PFOS |
| PZGS-GW-20-30-01-051324 | PFOS |
| MW18B-GW-225-235-01-050924 | PFNS |
| MW10A-GW-273-283-01-051324 | PFNS |
| MW18B-GW-225-235-01-050924 | N-MeFOSA |
| MW23A-GW-422-432-01-051324 | N-MeFOSA |
| PZHD-GW-21-26-01-051324 | N-MeFOSA |

N-MeFOSA in above samples, were qualified BJ for laboratory blank detection.

**Calibration (Section 2):** Calibration noncompliance are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/19/24 02:53 | ADONA | 182% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 02:53 | 11Cl-PF3OUdS | 142% | 70-130% | |
| CCV 06/19/24 06:41 | ADONA | 178% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 06:41 | 11Cl-PF3OUdS | 137% | 70-130% | |
| CCV 06/19/24 09:35 | ADONA | 176% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 09:35 | 11Cl-PF3OUdS | 136% | 70-130% | |
| CCV 06/19/24 11:49 | ADONA | 177% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/19/24 11:49 | 11Cl-PF3OUdS | 136% | 70-130% | |

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/19/24 02:53 | NFDHA | 41.6% | 70-130% | All samples, UJ |
| CCV 06/19/24 06:41 | NFDHA | 47.5% | 70-130% | |
| CCV 06/19/24 09:35 | NFDHA | 40.3% | 70-130% | |
| CCV 06/19/24 11:49 | NFDHA | 37.9% | 70-130% | |

**Blanks (Section 3Bii):** The PFOSA result for samples PZES-GW-6-11-01-051024 (L41364-39), PZFD-GW-33-38-01-051024 (L41364-40), PZFS-GW-18-28-01-051024 (L41364-41), PZGD-GW-35-40-01-051324 (L41364-42), PZHD-GW-21-26-01-051324 (L41364-44), PZHS-GW-6-16-01-051324 (L41364-45) and PZBD-GW-16-21-01-051324 (L41364-46) were within 10 times the laboratory blank concentration and qualified BJ.

The N-MeFOSA result for samples MW18B-GW-225-235-01-050924 (L41364-29), MW23A-GW-422-432-01-051324 (L41364-31) and PZHD-GW-21-26-01-051324 (L41364-44) were within 10 times the laboratory blank concentration and qualified BJ.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+) and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Five results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13A-GW-350-370-01-051024 | L41364-30 i | 13C4-PFBA | 5.28 | R |
| MW23A-GW-422-432-01-051324 | L41364-31 | 13C4-PFBA | 6.05 | R |
| MW23B-GW-299-309-01-051324 | L41364-32 | 13C4-PFBA | 8.78 | R |
| MW21A-GW-410-420-01-051324 | L41364-33 | 13C4-PFBA | 5.51 | R |
| MW17A-GW-230-240-01-051324 | L41364-34 | 13C4-PFBA | 12.5 | J+ |
| MW10A-GW-273-283-01-051324 | L41364-35 | 13C4-PFBA | 4.06 | R |
| MW23A-GW-422-432-01-051324 | L41364-31 | 13C2-8:2 FTS | 36.9 | UJ |
| MW23B-GW-299-309-01-051324 | L41364-32 | 13C2-8:2 FTS | 33.5 | UJ |
| MW10A-GW-273-283-01-051324 | L41364-35 | 13C2-8:2 FTS | 34.6 | UJ |
| MW10A-GW-273-283-01-051324 | L41364-35 | 13C3-HFPO-DA | 46.3 | UJ |
| MW10A-GW-273-283-01-051324 | L41364-35 | 13C5-PFPeA | 35.4 | J+ |
| PZFS-GW-18-28-01-051024 | L41364-41 | 13C3-HFPO-DA | 45.7 | UJ |
| MW10A-GW-273-283-01-051324 | L41364-35 | 13C8-PFOSA | 165 | None |
| MW10A-GW-273-283-01-051324 | L41364-35 | d7-NMe-FOSE | 179 | None |
| MW10A-GW-273-283-01-051324 | L41364-35 | d9-NEt-FOSE | 153 | None |

**Laboratory Control Sample (Section 5Bi):** Recoveries for the LCS are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS WG90106-102 | ADONA | 173% | 70-130% | ADONA was not detected, no qualification. |
| LCS WG90106-102 | NFDHA | 37.2% | 70-130% | UJ all samples. |
| LCS WG90106-103 | PFNA | 132% | 70-130% | Detections were qualified as estimated biased high (J+). |
| LCS WG90106-103 | 6:2 FTS | 143% | 70-130% | 6:2 FTS was not detected, no qualification. |
| LCS WG90106-103 | PFOSA | 139% | 70-130% | Detections previously qualified BJ due to laboratory contamination. |
| LCS WG90106-103 | 9Cl-PF3ONS | 139% | 70-130% | 9Cl-PF3ONS was not detected, no qualification. |
| LCS WG90106-103 | 11Cl-PF3OUdS | 141% | 70-130% | 11Cl-PF3OUdS was not detected, no qualification. |
| LCS WG90106-103 | ADONA | 203% | 70-130% | ADONA was not detected, no qualification. |
| LCS WG90106-103 | NFDHA | 46.9% | 70-130% | UJ all samples. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 4095

Work order number/Lab report ID: DPWG90486 (WG90106) Report date (mm/dd/yyyy): 7/3/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☐ | ☒ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | Yes, see data qualifications summary for details and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☐ | ☐ | ☒ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☐ | ☒ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis.**

**See attachment for additional comments and qualifications. Five results were rejected (R) due to severely low labeled analog (EIS) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Andrei Niculau Title: Chemist III

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90460 (WG90111)**

**Estimated Maximum Possible Concentration (EMPC):**

The following results were reported as an EMPC. These results were qualified as estimated (J).

| Sample | Analyte |
|---|---|
| MW26B-GW-249-259-01-051324 | PFPeS |
| MW26B-GW-249-259-01-051324 | PFOS |
| PZID-GW-22-27-01-051424 | PFOS |
| MW25B-GW-260-270-01-051324 | N-MeFOSA |
| MW26C-GW-130-140-01-051324 | N-MeFOSA |
| MW1B-GW-100-120-01-051324 | N-MeFOSA |
| Trip-Cooler1-051424 | N-MeFOSA |
| PZIS-GW-7-17-01-051424 | N-MeFOSA |
| MW26A-GW-380-390-01-051524 | N-MeFOSA |
| MW23C-GW-151-161-01-051724 | N-MeFOSA |

N-MeFOSA in above samples, were qualified BJ for laboratory blank detection.

**Calibration (Section 2):** Calibration noncompliance are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 06/18/24 03:48 | ADONA | 190% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/18/24 03:48 | 9Cl-PF3ONS | 135% | 70-130% | |
| CCV 06/18/24 03:48 | 11Cl-PF3OUdS | 147% | 70-130% | |
| CCV 06/18/24 07:36 | ADONA | 180% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/18/24 07:36 | 11Cl-PF3OUdS | 141% | 70-130% | |
| CCV 06/18/24 09:51 | ADONA | 179% | 70-130% | Results were not detected. No qualifiers. |
| CCV 06/18/24 09:51 | 11Cl-PF3OUdS | 140% | 70-130% | |
| CCV 06/18/24 03:48 | NFDHA | 36.3% | 70-130% | All samples, UJ |
| CCV 06/18/24 07:36 | NFDHA | 37.6% | 70-130% | |
| CCV 06/18/24 09:51 | NFDHA | 46.9% | 70-130% | |

**Blanks (Section 3Bii):** The N-MeFOSA result for samples MW25B-GW-260-270-01-051324 (L41364-51), MW26C-GW-130-140-01-051324 (L41364-53), MW1B-GW-100-120-01-051324 (L41364-55 N), PZIS-GW-7-17-01-051424 (L41393-2), MW26A-GW-380-390-01-051524 (L41393-6), and MW23C-GW-151-161-01-051724 (L41393-8) were within 10 times the trip blank concentration and qualified BJ for laboratory blank and trip cooler blank detection.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+) and detects associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW10B-GW-130-140-01-051324 | L41364-49 | 13C4-PFBA | 13.7 | J+ |
| MW15B-GW-215-225-01-051324 | L41364-50 | 13C4-PFBA | 3.21 | R |
| MW26B-GW-249-259-01-051324 | L41364-52 | 13C4-PFBA | 12.7 | J+ |
| MW1A-GW-360-370-01-051324 | L41364-54 | 13C4-PFBA | 4.68 | R |
| MW25A-GW-390-400-01-051524 | L41393-7 | 13C4-PFBA | 13.0 | J+ |
| MW23C-GW-151-161-01-051724 | L41393-8 | 13C4-PFBA | 46.4 | J+ |
| MW25B-GW-260-270-01-051324 | L41364-51 | 13C2-4:2 FTS | 178 | None |
| MW1A-GW-360-370-01-051324 | L41364-54 | 13C2-4:2 FTS | 151 | None |
| MW10B-GW-130-140-01-051324 | L41364-49 | 13C2-8:2 FTS | 44.9 | UJ |
| MW15B-GW-215-225-01-051324 | L41364-50 | 13C2-8:2 FTS | 32.2 | UJ |
| PZID-GW-22-27-01-051424 | L41393-3 | d9-NEt-FOSE | 155 | None |

**Laboratory Control Sample (Section 5Bi):** Recoveries for the LCS are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS/LCSD WG90111-102 WG90111-104 | ADONA | 181%/171% | 70-130% | ADONA was not detected, no qualification. |
| LCS/LCSD WG90111-102 | 9Cl-PF3ONS | 131% | 70-130% | 9Cl-PF3ONS was not detected, no qualification. |
| LCS/LCSD WG90111-102 | 11Cl-PF3OUdS | 132% | 70-130% | 11Cl-PF3OUdS was not detected, no qualification. |
| LCS/LCSD WG90111-102 WG90111-104 | NFDHA | 36.2%/36.6% | 70-130% | UJ all samples. |
| LCS WG90111-103 | PFOSA | 133% | 70-130% | Detects qualified J+. |
| LCS WG90111-103 | ADONA | 207% | 70-130% | ADONA was not detected, no qualification. |
| LCS WG90111-103 | 11Cl-PF3OUdS | 135% | 70-130% | 11Cl-PF3OUdS was not detected, no qualification. |

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| LCS WG90111-103 | 5:3 FTCA | 135% | 70-130% | 5:3 FTCA was not detected, no qualification. |
| LCS WG90111-103 | NFDHA | 35.7% | 70-130% | UJ all samples. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 4095

Work order number/Lab report ID: DPWG90460 (WG90111) Report date (mm/dd/yyyy): 6/28/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL, ICV and CCVs reviewed.<br>See data qualifications summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip-Cooler1-051424 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Yes, see data qualifications summary for details and qualifications. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | Yes, see data qualifications summary for details and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary for details and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☐ | ☐ | ☒ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☐ | ☒ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>LCS/LCSD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | 'PZBS-GW-6-11-01-051324' and 'PZID-GW-22-27-01-051424' (SGS AXYS ID: L41364-48 and L41393-3, respectively) had elevated limits as not all the sample passed through the SPE column during sample prep due to matrix. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis.**

**See attachment for additional comments and qualifications. Two results were rejected (R) due to severely low labeled analog (EIS) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Andrei Niculau Title: Chemist III

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90481 (WG90140)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 6/17/24 19:05 | ADONA | 193% | 70-130% | ADONA, 9Cl-PF3ONS, and 11Cl-PF3OUdS results were not detected and were acceptable without qualification. NFDHA results were not detected and qualified UJ. |
| CCV 6/17/24 19:05 | 9Cl-PF3ONS | 137% | 70-130% | |
| CCV 6/17/24 19:05 | 11Cl-PF3OUdS | 145% | 70-130% | |
| CCV 6/17/24 19:05 | NFDHA | 34.8% | 70-130% | |
| CCV 6/17/24 22:53 | NFDHA | 30.7% | 70-130% | NFDHA results were not reported from this CCV. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below. Seven results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW27B-GW-120-130-01-051424 | L41378-2 | 13C4-PFBA | 20.2 | J+ |
| MW27A-GW-221-222-01-051424 | L41378-3 | 13C4-PFBA | 14.1 | J+ |
| MW20B-GW-90-100-01-051424 | L41378-4 | 13C4-PFBA | 2.11 | R |
| MW20B-GW-90-100-01-051424 | L41378-4 | 13C5-PFPeA | 26.0 | J+ |
| MW20B-GW-90-100-01-051424 | L41378-4 | 13C3-HFPO-DA | 45.6 | UJ |
| OW20J-1-GW-310-320-01-051424 | L41378-5 | 13C4-PFBA | 8.32 | R |
| OW20J-1-GW-310-320-01-051424 | L41378-5 | 13C8-PFOSA | 152 | No Qualifier |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | 13C4-PFBA | 7.19 | R |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | 13C2-8:2 FTS | 44.6 | UJ |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | 13C8-PFOSA | 168 | No Qualifier |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | d7-NMe-FOSE | 159 | No Qualifier |
| MW5A-GW-210-220-01-051424 | L41378-7 | 13C4-PFBA | 1.05 | R |
| MW5A-GW-210-220-01-051424 | L41378-7 | 13C5-PFPeA | 7.52 | R |
| MW5A-GW-210-220-01-051424 | L41378-7 | 13C8-PFOSA | 172 | No Qualifier |
| MW5A-GW-210-220-01-051424 | L41378-7 | d7-NMe-FOSE | 174 | No Qualifier |
| MW5A-GW-210-220-01-051424 | L41378-7 | d9-NEt-FOSE | 155 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW5A-GW-210-220-01-051424 | L41378-7 | 13C3-HFPO-DA | 30.2 | UJ |
| MW5B-GWQC-BLANK-110-120-01-051424 | L41378-8 | 13C4-PFBA | 7.68 | R |
| MW5B-GWQC-BLANK-110-120-01-051424 | L41378-8 | 13C2-4:2 FTS | 163 | No Qualifier |
| MW5B-GWQC-BLANK-110-120-01-051424 | L41378-8 | d9-NEt-FOSE | 151 | No Qualifier |
| MW5B-GWQC-BLANK-110-120-01-051424 | L41378-8 | 13C3-HFPO-DA | 49.1 | UJ |
| MW4A-GW-140-160-01-051424 | L41378-9 | 13C4-PFBA | 6.54 | R |
| MW4A-GW-140-160-01-051424 | L41378-9 | 13C2-4:2 FTS | 186 | No Qualifier |
| OW50-1-GW-200-210-01-051424 | L41378-10 | 13C4-PFBA | 10 | J+ |
| OW50-1-GW-200-210-01-051424 | L41378-10 | 13C2-4:2 FTS | 167 | No Qualifier |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LCS/LCSD noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG90140-102 /WG90140-104 | ADONA | **186/177** | No Qualifier |
| | 5:3 FTCA | 128/**131** | No Qualifier |
| | NFDHA | **36.2/37.0** | UJ |
| WG90140-103 | 6:2 FTS | **142** | No Qualifier |
| | ADONA | **208** | No Qualifier |
| | 9Cl-PF3ONS | **141** | No Qualifier |
| | 11Cl-PF3OUdS | **145** | No Qualifier |
| | 3:3 FTCA | **132** | No Qualifier |
| | 5:3 FTCA | **134** | No Qualifier |
| | NFDHA | **44.8** | UJ |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW27B-GW-120-130-01-051424 | L41378-2 | PFOS |
| MW20B-GW-90-100-01-051424 | L41378-4 | PFOS |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | PFPeS |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | PFOS |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | PFNS |
| OW20J-3-GW-280-290-01-051424 | L41378-6 | N-MeFOSA |
| MW5A-GW-210-220-01-051424 | L41378-7 | PFOS |
| MW5B-GWQC-BLANK-110-120-01-051424 | L41378-8 | N-MeFOSA |

Note that an EQuIS EDD was not received for this data set and the results are not included in the database.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90481 (WG90140) Report date (mm/dd/yyyy): 6/27/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Sample receipt temperature was 12.6˚C, and above the 6˚C limit. Results were qualified as estimated (UJ and J-). |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip-cooler1-051524 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☐ | ☒ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☐ | ☒ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>LCS/LCSD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications. Seven results were rejected (R) due to severely low labeled analog recoveries.**

**Note that an EQuIS EDD was not received for this data set and the results are not included in the database.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG90783 (WG90250)**

**Matrix spike/ Matrix spike duplicates – (Section 6AV):** Results associated with high recoveries were nondetects and were acceptable without qualification.

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90250-105 (MS) | **ADONA** | **131%** | 70-130% | No qualifiers |
| WG90250-106 (MSD) | **PFOS*** | **63.7%** | 70-130% | No qualifiers |

* MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines.).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG90783 (WG90250) Report date (mm/dd/yyyy): 7/29/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample PZED-GW-16-21-03-051024 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See attachment for details. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate:<br>PZED-GW-16-21-01-051024 / PZED-GW-16-21-02-051024 / PZED-GW-16-21-03-051024 (RPDs ok) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG91375 (WG90971)**

**Calibration (Section 2): Calibration** noncompliance's are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/07/24 00:45 | PFEESA | **131** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 9/07/24 04:49 | PFEESA | **131** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |

**Laboratory control spike/ Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ and J-). Results associated with high recoveries were nondetects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90971-102 | 3:3 FTCA | **140%** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG89882-102 | N-EtFOSA | **134%** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG90971-103 | 3:3 FTCA | **139%** | 70-130% | All the associated sample results were not detected. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WC-6A-SOIL-3-5-01-072224 | L41739-1 | D3-N-MeFOSA | 43.9 | UJ |
| WC-6A-SOIL-3-5-01-072224 | L41739-1 | D5-N-EtFOSA | 37.4 | UJ |
| WC-6A-SOIL-14-16-01-072224 | L41739-2 | D5-N-EtFOSA | 36.4 | UJ |
| WC-6A-SOIL-35-37-01-072224 | L41739-3 | D3-N-MeFOSA | 35.1 | UJ |
| WC-6A-SOIL-35-37-01-072224 | L41739-3 | D5-N-EtFOSA | 18.8 | UJ |
| WC-6A-SOIL-56-58-01-072224 | L41739-4 | D3-N-MeFOSA | 33.3 | UJ |
| WC-6A-SOIL-56-58-01-072224 | L41739-4 | D5-N-EtFOSA | 16.4 | UJ |
| WC-6A-SOIL-68-70-01-072224 | L41739-5 | D3-N-MeFOSA | 37.3 | UJ |
| WC-6A-SOIL-68-70-01-072224 | L41739-5 | D5-N-EtFOSA | 26.6 | UJ |
| WC-6A-SOIL-77-79-01-072224 | L41739-6 | D3-N-MeFOSA | 42.4 | UJ |
| WC-6A-SOIL-77-79-01-072224 | L41739-6 | D5-N-EtFOSA | 27.7 | UJ |
| WC-6A-SOIL-77-79-01-072224 (Duplicate) | WG90971-104 (DUP L41739-6) | D5-N-EtFOSA | 43.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WC-6A-SOIL-85-87-01-072224 | L41739-7 | D3-N-MeFOSA | 42.1 | UJ |
| WC-6A-SOIL-85-87-01-072224 | L41739-7 | D5-N-EtFOSA | 23.9 | UJ |
| WC-6A-SOIL-85-87-02-072224 | L41739-8 | D3-N-MeFOSA | 41.6 | UJ |
| WC-6A-SOIL-85-87-02-072224 | L41739-8 | D5-N-EtFOSA | 22.3 | UJ |
| WC-5A-SOIL-5-6-01-072524 | L41739-9 | D3-N-MeFOSA | 46.5 | UJ |
| WC-5A-SOIL-5-6-01-072524 | L41739-9 | D5-N-EtFOSA | 32.6 | J- |
| WC-5A-SOIL-16-18-01-072524 | L41739-10 | D5-N-EtFOSA | 38.3 | UJ |
| WC-5A-SOIL-16-18-01-072524 | L41739-10 | D3-N-MeFOSA | 53.2 | UJ |
| WC-5A-SOIL-20-22-01-072524 | L41739-11 | D3-N-MeFOSA | 38.5 | UJ |
| WC-5A-SOIL-20-22-01-072524 | L41739-11 | D5-N-EtFOSA | 21.8 | UJ |
| WC-5A-SOIL-31-33-01-072524 | L41739-12 | D5-N-EtFOSA | 38.8 | UJ |
| WC-5A-SOIL-47-49-01-072524 | L41739-13 | D3-N-MeFOSA | 36.5 | UJ |
| WC-5A-SOIL-47-49-01-072524 | L41739-13 | D5-N-EtFOSA | 20.6 | UJ |
| WC-5A-SOIL-56-58-01-072524 | L41739-14 | D3-N-MeFOSA | 42.6 | UJ |
| WC-5A-SOIL-56-58-01-072524 | L41739-14 | D5-N-EtFOSA | 30.8 | UJ |
| WC-5A-SOIL-64-66-01-072524 | L41739-15 | D3-N-MeFOSA | 38.5 | UJ |
| WC-5A-SOIL-64-66-01-072524 | L41739-15 | D5-N-EtFOSA | 20.7 | UJ |
| WC-5A-SOIL-64-66-02-072524 | L41739-16 | D3-N-MeFOSA | 32.3 | UJ |
| WC-5A-SOIL-64-66-02-072524 | L41739-16 | D5-N-EtFOSA | 15.8 | UJ |
| WC-5A-SOIL-76-78-01-072524 | L41739-17 | D3-N-MeFOSA | 43.3 | UJ |
| WC-5A-SOIL-76-78-01-072524 | L41739-17 | D5-N-EtFOSA | 24.8 | UJ |
| WC-5A-SOIL-87-89-01-072524 | L41739-18 | D3-N-MeFOSA | 43.1 | UJ |
| WC-5A-SOIL-87-89-01-072524 | L41739-18 | D5-N-EtFOSA | 25.8 | UJ |
| WC-5A-SOIL-98-100-01-072524 | L41739-19 | D3-N-MeFOSA | 38.3 | UJ |
| WC-5A-SOIL-98-100-01-072524 | L41739-19 | D5-N-EtFOSA | 28.7 | UJ |
| WC-5A-SOIL-98-100-02-072524 | L41739-20 | D3-N-MeFOSA | 35.3 | UJ |
| WC-5A-SOIL-98-100-02-072524 | L41739-20 | D5-N-EtFOSA | 26.1 | UJ |

**Field and Laboratory Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/g) | Field Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **WC-5A-SOIL-64-66-01-072524/WC-5A-SOIL-64-66-02-072524:** | | | |
| PFBA | 6.26 | 21.3 | **109** |
| PFOA | 0.423 | 1.1 | **89** |

| Analyte | Parent Sample Result (ng/g) | Lab Duplicate Result (ng/g) | RPD (%) |
|---|---|---|---|
| **WC-6A-SOIL-77-79-01-072224/WC-6A-SOIL-77-79-01-072224 (Duplicate):** | | | |
| PFOS | 0.091 | 0.065 | **33.4** |

**Matrix spike/ Matrix spike duplicates – (Section 6AV) ;).** Results associated with high recoveries were nondetects and were acceptable without qualification. MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines.).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90971-105 (MS) | 3:3 FTCA | **137%** | 70-130% | No qualifiers |
| WG90971-106 (MSD) | 3:3 FTCA | **132%** | 70-130% | No qualifiers |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91375 (WG90971) Report date (mm/dd/yyyy): 9/27/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD performed on project sample WC-6A-SOIL-77-79-01-072224 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☒ | ☐ | ☐ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicates:<br>WC-6A-SOIL-85-87-01-072224 / WC-6A-SOIL-85-87-02-072224<br>WC-5A-SOIL-64-66-01-072524 / WC-5A-SOIL-64-66-02-072524<br>WC-5A-SOIL-98-100-01-072524 / WC-5A-SOIL-98-100-02-072524 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 50% RPD limit, or ± 5xRL, used for soil matrix.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**Results for five compounds (all samples) are rejected (R) due to severely low OPR (LCS/LCSD) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG91338 (WG90972)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WC-5A-SOIL-101-103-01-072524 | L41739-21 | 13C4-PFBA | 17 | J+ |
| WC-5A-SOIL-101-103-01-072524 | L41739-21 | D5-N-EtFOSA | 37.5 | UJ |
| WC-5A-SOIL-101-103-01-072524 | L41739-22 | 13C4-PFBA | 20.7 | J+ |
| WC-5A-SOIL-101-103-01-072524 | L41739-22 | D3-N-MeFOSA | 38.9 | UJ |
| WC-5A-SOIL-101-103-01-072524 | L41739-22 | D5-N-EtFOSA | 23.7 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG90972-103 | PFDoS | 69.5 | UJ |

**Matrix spike / Matrix spike duplicates (Section 5):** MS/MSD noncompliances are summarized below.

| Sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WC-5A-SOIL-118-119-01-072524 | PFBA | **51.3**/81.9 | J- |

**10. Additional comments about lab report:** Results with both high and low bias are qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91338 (WG90972) Report date (mm/dd/yyyy): 9/18/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/13/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | WC-5A-SOIL-118-119-01-072524 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/13/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPW91282 (WG90981)**

**Chain of custody, preservation and holding time (Section 1.E):**

Samples Trip-Cooler1-071624, Field Blank-071624, EQ-HYDRO-071624, EQ-PERI-071624, EQ-DIPPER-071624, WC6-WAT-BULK-01-071624, EP26D-WAT-BULK-01-071624, WC3-WAT-BULK-01-071624, and WC2-WAT-BULK-01-071624 receipt temperature (7.2°C), Trip-Cooler3-071624 receipt temperature (10.4°C), and Trip-Cooler4-071624 receipt temperature (7.5°C) did not meet the required criteria (< 6°C). Results for sample received at temperatures greater than 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects.

**Laboratory control spike/ Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ) for the non detects. Results associated with high recoveries were nondetects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90981-102 /103 | 3:3 FTCA | **135/**124 | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG90981-102 /103 | PFDS | 79.4**/69.3** | 70-130% | Results associated with low recoveries were qualified as estimated (UJ). |
| WG90981-102 /103 | PFDoS | 72.9**/49.4** | 70-130% | Results associated with low recoveries were qualified as estimated (UJ). |
| WG90981-102 /103 | 11Cl-PF3OUdS | 78.7**/64.2** | 70-130% | Results associated with low recoveries were qualified as estimated (UJ). |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Trip-Cooler1-071624 | L41686-1 | D3-N-MeFOSA | 29.4 | UJ |
| Trip-Cooler1-071624 | L41686-1 | D5-N-EtFOSA | 27.3 | UJ |
| Trip-Cooler1-071624 | L41686-1 | d7-NMe-FOSE | 49.8 | UJ |
| Trip-Cooler2-071624 | L41686-2 | D3-N-MeFOSA | 34.1 | UJ |
| Trip-Cooler2-071624 | L41686-2 | D5-N-EtFOSA | 26.7 | UJ |
| Trip-Cooler3-071624 | L41686-3 | D3-N-MeFOSA | 32.9 | UJ |
| Trip-Cooler3-071624 | L41686-3 | D5-N-EtFOSA | 27.5 | UJ |
| Trip-Cooler4-071624 | L41686-4 | D3-N-MeFOSA | 34.2 | UJ |
| Trip-Cooler4-071624 | L41686-4 | D5-N-EtFOSA | 25.4 | UJ |
| Trip-Cooler4-071624 | L41686-4 | d7-NMe-FOSE | 48.0 | UJ |
| Trip-Cooler4-071624 | L41686-4 | d9-NEt-FOSE | 49.0 | UJ |
| Field Blank-071624 | L41686-5 | D3-N-MeFOSA | 33.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Field Blank-071624 | L41686-5 | D3-N-MeFOSA | 26.5 | UJ |
| EQ-HYDRO-071624 | L41686-6 | D3-N-MeFOSA | 37.7 | UJ |
| EQ-HYDRO-071624 | L41686-6 | D5-N-EtFOSA | 28.4 | UJ |
| EQ-PERI-071624 | L41686-7 | D3-N-MeFOSA | 31.1 | UJ |
| EQ-PERI-071624 | L41686-7 | D5-N-EtFOSA | 24.4 | UJ |
| EQ-PERI-071624 | L41686-7 | d7-NMe-FOSE | 47.8 | UJ |
| EQ-PERI-071624 | L41686-7 | d9-NEt-FOSE | 49.1 | UJ |
| EQ-DIPPER-071624 | L41686-8 | D3-N-MeFOSA | 38.5 | UJ |
| EQ-DIPPER-071624 | L41686-8 | D5-N-EtFOSA | 31.8 | UJ |
| WC6-WAT-BULK-01-071624 | L41686-9 | 13C4-PFBA | 12.6 | J+ |
| WC6-WAT-BULK-01-071624 | L41686-9 | 13C2-PFTeDA | 36.0 | UJ<br>PFTrDA: UJ |
| EP26D-WAT-BULK-01-071624 | L41686-10 | 13C4-PFBA | 10.3 | J+ |
| EP26D-WAT-BULK-01-071624 | L41686-10 | 13C2-PFTeDA | 46.6 | UJ<br>PFTrDA: UJ |
| WC3-WAT-BULK-01-071624 | L41686-11 | 13C2-PFTeDA | 40.4 | UJ<br>PFTrDA: UJ |
| WC3-WAT-BULK-01-071624 | L41686-11 | D5-N-EtFOSA | 42.6 | UJ |
| WC2-WAT-BULK-01-071624 | L41686-12 | 13C4-PFBA | 18.5 | J+ |
| WC2-WAT-BULK-01-071624 | L41686-12 | 13C2-PFTeDA | 31.4 | UJ<br>PFTrDA: UJ |
| WC2-WAT-BULK-01-071624 | L41686-12 | D5-N-EtFOSA | 47.1 | UJ |
| WC2-WAT-BULK-01-071624 | L41686-12 | d9-NEt-FOSE | 49.5 | UJ |
| SP4-GW-116-126-01-071524 | L41686-13 | 13C2-PFTeDA | 39.6 | UJ<br>PFTrDA: UJ |
| SP4-GW-116-126-01-071524 | L41686-13 | D5-N-EtFOSA | 40.9 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | 13C2-PFDoA | 49.9 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | 13C2-PFTeDA | 40.0 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | D3-N-MeFOSA | 39.1 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | D5-N-EtFOSA | 30.6 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | D3-MeFOSAA | 47.9 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | d7-NMe-FOSE | 45.1 | UJ |
| PL41-GW-72-82-01-071524 | L41686-14 | d9-NEt-FOSE | 44.3 | UJ |
| OD3-WAT-BULK-01-071524 | L41686-15 | 13C2-PFTeDA | 29.9 | UJ<br>PFTrDA: UJ |
| OD3-WAT-BULK-01-071524 | L41686-15 | D3-N-MeFOSA | 47.6 | UJ |
| OD3-WAT-BULK-01-071524 | L41686-15 | D5-N-EtFOSA | 39.7 | UJ |
| OD3-WAT-BULK-01-071524 | L41686-15 | d7-NMe-FOSE | 46.5 | UJ |
| OD3-WAT-BULK-01-071524 | L41686-15 | d9-NEt-FOSE | 42.5 | UJ |
| PL41-GW-72-82-02-071524 | L41686-16 | 13C2-PFTeDA | 40.2 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-02-071524 | L41686-16 | D3-N-MeFOSA | 34.6 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PL41-GW-72-82-02-071524 | L41686-16 | D5-N-EtFOSA | 29.8 | UJ |
| PL41-GW-72-82-02-071524 | L41686-16 | D3-MeFOSAA | 49.1 | UJ |
| PL41-GW-72-82-02-071524 | L41686-16 | d7-NMe-FOSE | 48.4 | UJ |
| PL41-GW-72-82-02-071524 | L41686-16 | d9-NEt-FOSE | 48.9 | UJ |
| PL41-GW-72-82-03-071524 | L41686-17 (A) | 13C2-PFTeDA | 32.5 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-03-071524 | L41686-17 (A) | D3-N-MeFOSA | 43.8 | UJ |
| PL41-GW-72-82-03-071524 | L41686-17 (A) | D5-N-EtFOSA | 35.3 | UJ |
| PL41-GW-72-82-03-071524 | L41686-17 (A) | d7-NMe-FOSE | 47.6 | UJ |
| PL41-GW-72-82-03-071524 | L41686-17 (A) | d9-NEt-FOSE | 45.8 | UJ |

**Matrix spike/ Matrix spike duplicates – (Section 6AV):** Results associated with low recoveries were qualified and estimated as (UJ).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90981-104(MS)/105 (MSD) | PFHxA * | **16.0/53.9** | 70-130% | No qualifiers |
| WG90981-104(MS)/105 (MSD) | PFDS | **61.4/66.7** | 70-130% | UJ |
| WG90981-104(MS)/105 (MSD) | PFDoS | **51.8/56.3** | 70-130% | UJ |
| WG90981-104(MS)/105 (MSD) | 11Cl-PF3OUdS | **52.6/58.3** | 70-130% | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91282 (WG90981) Report date (mm/dd/yyyy): 9/18/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/18/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | Samples were analyzed within the SGS AXYS extended-time storage time limit of 90 days. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-Cooler1-071624<br>Trip-Cooler2-071624<br>Trip-Cooler3-071624<br>Trip-Cooler4-071624<br>Field Blank-071624<br>EQ-HYDRO-071624<br>EQ-PERI-071624<br>EQ-DIPPER-071624 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | MS/MSD performed on project sample PL41-GW-72-82-03-071524 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>FIeld Triplicate:<br>PL41-GW-72-82-01-071524/PL41-GW-72-82- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 02-071524/PL41-GW-72-82-03-071524 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/18/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG91390 (WG90982)**

**Chain of custody, preservation and holding time (Section 1.E):**

Samples EP16-WAT-BULK-01-071624, WC5-WAT-BULK-01-071624, WL11-WAT-BULK-01-071624, WC4-WAT-BULK-01-071624, WC1-WAT-BULK-01-071624, RC2-WAT-BULK-01-071624, and RC2-WAT-BULK-02-071624 receipt temperature (10.4°C), BL5-WAT-BULK-01-071624, BL3-WAT-BULK-01-071624, BL5-WAT-BULK-02-071624, RC3-WAT-BULK-01-071624, RC5-WAT-BULK-01-071624, RC5-WAT-BULK-02-071624, RC5-WAT-BULK-03-071624 and RC23-WAT-BULK-01-071624 receipt temperature (7.3°C) did not meet the required criteria (< 6°C). Results for sample received at temperatures greater than 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects.

**Blanks-(Section 3Aii):**

The analyte PFOSA (0.914 ng/L) was detected in method blank. Sample results did not require qualification based on the associated blank if the sample result was reported as not detected or was detected at a concentration above the laboratory reporting limit and significantly higher than the associated blank concentration. Data were qualified B/BJ if the associated sample result was within 10 times the blank concentration.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Six results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD4-WAT-BULK-01-071524 | L41686-18 | 13C2-4:2 FTS | 187 | No Qualifier |
| OD4-WAT-BULK-01-071524 | L41686-18 | 13C4-PFBA | 26.6 | J+ |
| OD5-WAT-BULK-01-071524 | L41686-19 | 13C4-PFBA | 47.8 | J+ |
| EP16-WAT-BULK-01-071624 | L41686-20 | 13C4-PFBA | 22.6 | J+ |
| EP16-WAT-BULK-01-071624 | L41686-20 | 13C7-PFUnA | 48.7 | UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | 13C2-PFDoA | 39.3 | UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | 13C2-PFTeDA | 21.1 | UJ<br>PFTrDA: UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | 13C8-PFOS | 30.5 | J+ |
| EP16-WAT-BULK-01-071624 | L41686-20 | D3-N-MeFOSA | 48.0 | UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | D5-N-EtFOSA | 46.2 | UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | d7-NMe-FOSE | 29.3 | UJ |
| EP16-WAT-BULK-01-071624 | L41686-20 | d9-NEt-FOSE | 21.3 | UJ |
| WL11-WAT-BULK-01-071624 | L41686-22 | 13C4-PFBA | 33.2 | J+ |
| WL11-WAT-BULK-01-071624 | L41686-22 | 13C2-PFTeDA | 8.88 | R<br>PFTrDA: R |
| WL11-WAT-BULK-01-071624 | L41686-22 | d7-NMe-FOSE | 12.2 | UJ |
| WL11-WAT-BULK-01-071624 | L41686-22 | d9-NEt-FOSE | 4.79 | R |
| WC4-WAT-BULK-01-071624 | L41686-23 | 13C2-4:2 FTS | 157 | No Qualifier |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WC1-WAT-BULK-01-071624 | L41686-24 | 13C4-PFBA | 10.3 | J+ |
| RC2-WAT-BULK-01-071624 | L41686-25 | 13C4-PFBA | 8.80 | R |
| RC2-WAT-BULK-02-071624 | L41686-26 | 13C4-PFBA | 6.29 | R |
| BL5-WAT-BULK-01-071624 | L41686-27 | 13C4-PFBA | 8.47 | R |
| BL3-WAT-BULK-01-071624 | L41686-28 | 13C4-PFBA | 15.6 | J+ |
| BL5-WAT-BULK-02-071624 | L41686-29 | 13C4-PFBA | 24.8 | J+ |
| BL5-WAT-BULK-02-071624 | L41686-29 | 13C2-PFTeDA | 31.8 | UJ<br>PFTrDA: UJ |
| BL5-WAT-BULK-02-071624 | L41686-29 | d7-NMe-FOSE | 41.6 | UJ |
| BL5-WAT-BULK-02-071624 | L41686-29 | d9-NEt-FOSE | 25.9 | UJ |
| RC23-WAT-BULK-01-071624 | L41686-34 | 13C4-PFBA | 21.8 | J+ |
| RC23-WAT-BULK-01-071624 | L41686-34 | 13C2-4:2 FTS | 154 | No Qualifier |

**Matrix spike/ Matrix spike duplicates – (Section 6AV).** *MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

| MS/MSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG90982-104 (MS)/105 (MSD) | **PFOS *** | **162**/84.2 | 70-130% | No qualifiers |

**Field Duplicates (Section 6Bi):**

Results qualified as estimated (J) due to field imprecision are listed below.

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | RPD (%)/absolute difference |
|---|---|---|---|
| **BL5-WAT-BULK-01-071624/BL5-WAT-BULK-02-071624:** | | | |
| PFMPA | 2.48 | 5.46 | >±RL |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| WC4-WAT-BULK-01-071624 | L41686-23 | PFHxS |
| WC1-WAT-BULK-01-071624 | L41686-24 | PFHpS |

Results associated with both high and low bias were qualified as J (no bias flag).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91260 (WG90983) Report date (mm/dd/yyyy): 9/17/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/18/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for templerature exceedances and qualifications. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed.See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-cooler1-071824<br>Trip-cooler2-071824<br>Trip-cooler3-071824<br>Trip-cooler4-071824<br>Trip-cooler5-071824 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☒ | ☐ | ☐ | Detects were greater than the RL<br>PFOSA 0.423 ng/L in Trip-cooler2-071824 |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | Detects were greater than the RL<br>6:2 FTS 1.85 J ng/L in Method blank |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. Results for eight compounds are rejected (R) due to severely low recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☒ | ☐ | MS/MSD performed on project sample MW28C-GW-70-80-03-081324 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>MW14D-GW-6-21-01-071724/MW14D-GW-6-21-02-071724 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | Field Triplicate:<br>MW28C-GW-70-80-01-081324/MW28C-GW-70-80-02-081324/MW28C-GW-70-80-03-081324 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% RPD limit, or ± RL, used for water matrix.<br>MSD RPDs ok<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample size used for four samples. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**Results for eight compounds are rejected (R) due to severely low labelled analog (Surrogate) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Kiranmayi G Title: Environmental Chemist (III)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/18/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG91319 (WG90984)**

**Chain of custody, preservation and holding time (Section 1.E):**

Six samples had a receipt temperature of 6.2°C and five samples had a receipt temperature of 11.3°C, and were above the criteria of 6°C. Results for samples received at temperatures greater that 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects.

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below. Six results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW2D-GW-7-17-01-071724 | L41704-17 | 13C4-PFBA | 40.7 | J+ |
| MW2D-GW-7-17-02-071724 | L41704-18 | 13C4-PFBA | 42.6 | J+ |
| MW8B-GW-50-60-03-071724 | L41704-21 | 13C4-PFBA | 44.3 | J+ |
| MW6C-GW-35-40-01-071824 | L41704-28 | 13C4-PFBA | 48.4 | J+ |
| MW6D-GW-8-18-01-071824 | L41704-29 | 13C4-PFBA | 38.2 | J+ |
| EQ-GE054B-071824 | L41704-31 | D3-N-MeFOSA | 48.1 | UJ |
| EQ-GE054B-071824 | L41704-31 | D5-N-EtFOSA | 47.4 | UJ |
| MW25B-GW-260-270-01-071824 | L41704-32 | 13C4-PFBA | 2.39 | R |
| MW25B-GW-260-270-01-071824 | L41704-32 | 13C5-PFPeA | 19.2 | J+ |
| MW25A-GW-390-400-01-071824 | L41704-33 | 13C4-PFBA | 7.42 | R |
| MW20A-GW-130-140-01-071824 | L41704-34 | 13C4-PFBA | 7.47 | R |
| Site J-GW-358-368-01-071824 | L41704-35 | 13C4-PFBA | 7.13 | R |
| Site I-GW-411-421-01-071824 | L41704-36 | 13C4-PFBA | 5.23 | R |
| MW23C-GW-151-161-01-071824 | L41704-37 | 13C4-PFBA | 2.78 | R |
| MW23C-GW-151-161-01-071824 | L41704-37 | 13C5-PFPeA | 35.0 | J+ |
| Trip-Cooler1-081524 | L41852-1 | D5-N-EtFOSA | 49.9 | UJ |
| MW28B-GW-119-129-01-081324 | L41852-2 | D3-N-MeFOSA | 42.8 | UJ |
| MW28B-GW-119-129-01-081324 | L41852-2 | D5-N-EtFOSA | 39.6 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LCS noncompliances are summarized below.

| LCS ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG90984-102/ WG90984-103 | 3:3 FTCA | 145/141 | No qualifiers |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| MW8B-GW-50-60-03-071724 | 3:3 FTCA | 135/136 | No qualifier |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW20A-GW-130-140-01-071824 | L41704-34 | PFNS |
| MW23C-GW-151-161-01-071824 | L41704-37 | PFOS |
| MW25B-GW-260-270-01-071824 | L41704-32 | PFOS |
| MW25B-GW-260-270-01-071824 | L41704-32 | PFHpA |
| MW25A-GW-390-400-01-071824 | L41704-33 | PFNS |
| MW25A-GW-390-400-01-071824 | L41704-35 | PFNS |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91319 (WG90984) Report date (mm/dd/yyyy): 9/18/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/18/2026

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip-Cooler1-081524<br>EQ-GE054B-071824 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. Results for six compounds are rejected (R) due to severely low recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | . |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MW8B-GW-50-60-03-071724 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Duplicate:<br>MW2D-GW-7-17-01-071724/MW2D-GW-7-17-02-071724<br>Field Triplicate:<br>MW8B-GW-50-60-01-071724/MW8B-GW-50-60-02-071724/MW8B-GW-50-60-03-071724 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? | | ☒ | ☐ | ☐ | RPDs ok |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used for one sample |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**Results for six compounds are rejected (R) due to severely low surrogate recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/18/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG91385 (WG90985)**

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 9/9/24 18:07 | 3:3 FTCA | 134% | 70-130% | 3:3 FTCA results were nondetect and were acceptable without qualification. |
| CCV 9/9/24 22:13 | 3:3 FTCA | 131% | 70-130% | |
| CCV 9/11/24 17:01 | 3:3 FTCA | 132% | 70-130% | 3:3 FTCA and PFEESA results were not reported from these CCVs. No qualifiers. |
| CCV 9/11/24 17:01 | PFEESA | 132% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PZED-GW-16-21-01-071824 | L41704-24 | 13C2-PFTeDA | 36.2 | UJ PFTrDA: UJ |
| PZED-GW-16-21-01-071824 | L41704-24 | D3-N-MeFOSA | 48.5 | UJ |
| PZED-GW-16-21-01-071824 | L41704-24 | D5-N-EtFOSA | 37.8 | UJ |
| PZED-GW-16-21-02-071824 | L41704-25 | 13C2-PFTeDA | 36.8 | UJ PFTrDA: UJ |
| PZED-GW-16-21-02-071824 | L41704-25 | D3-N-MeFOSA | 40.1 | UJ |
| PZED-GW-16-21-02-071824 | L41704-25 | D5-N-EtFOSA | 32.4 | UJ |
| PZED-GW-16-21-03-071824 | L41704-26 (A) | 13C2-PFTeDA | 45.5 | UJ PFTrDA: UJ |
| PZED-GW-16-21-03-071824 | L41704-26 (A) | D3-N-MeFOSA | 49.8 | UJ |
| PZED-GW-16-21-03-071824 | L41704-26 (A) | D5-N-EtFOSA | 41.3 | UJ |
| MW8A-GW-202-212-01-071824 | L41704-38 | 13C2-PFTeDA | 40.4 | UJ PFTrDA: UJ |
| MW8A-GW-202-212-01-071824 | L41704-38 | D3-N-MeFOSA | 36.1 | UJ |
| MW8A-GW-202-212-01-071824 | L41704-38 | D5-N-EtFOSA | 25.5 | UJ |
| MW8A-GW-202-212-01-071824 | L41704-38 | d7-NMe-FOSE | 41.7 | UJ |
| MW8A-GW-202-212-01-071824 | L41704-38 | d9-NEt-FOSE | 37.4 | UJ |
| MW23B-GW-299-309-01-071824 | L41704-39 | 13C4-PFBA | 30.7 | J+ |
| MW23B-GW-299-309-01-071824 | L41704-39 | 13C2-PFTeDA | 47.5 | UJ PFTrDA: UJ |
| MW23B-GW-299-309-01-071824 | L41704-39 | D5-N-EtFOSA | 49.8 | UJ |
| MW23B-GW-299-309-01-071824 | L41704-39 | d7-NMe-FOSE | 48.0 | UJ |
| MW23B-GW-299-309-01-071824 | L41704-39 | d9-NEt-FOSE | 41.5 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW26A-GW-380-390-01-071824 | L41704-40 | 13C4-PFBA | 33.4 | J+ |
| MW26A-GW-380-390-01-071824 | L41704-40 | 13C2-PFTeDA | 46.8 | UJ<br>PFTrDA: UJ |
| MW26A-GW-380-390-01-071824 | L41704-40 | D5-N-EtFOSA | 42.3 | UJ |
| MW26A-GW-380-390-01-071824 | L41704-40 | d7-NMe-FOSE | 45.2 | UJ |
| MW26A-GW-380-390-01-071824 | L41704-40 | d9-NEt-FOSE | 40.5 | UJ |
| MW26C-GW-130-140-01-071824 | L41704-41 | 13C2-PFTeDA | 39.3 | UJ<br>PFTrDA: UJ |
| MW26C-GW-130-140-01-071824 | L41704-41 | D3-N-MeFOSA | 33.0 | UJ |
| MW26C-GW-130-140-01-071824 | L41704-41 | D5-N-EtFOSA | 26.6 | UJ |
| MW26C-GW-130-140-01-071824 | L41704-41 | d7-NMe-FOSE | 33.3 | UJ |
| MW26C-GW-130-140-01-071824 | L41704-41 | d9-NEt-FOSE | 30.3 | UJ |
| MW10A-GW-273-283-01-071824 | L41704-42 | 13C4-PFBA | 19.8 | J+ |
| MW10A-GW-273-283-01-071824 | L41704-42 | 13C2-PFTeDA | 43.1 | UJ<br>PFTrDA: UJ |
| MW10A-GW-273-283-01-071824 | L41704-42 | D5-N-EtFOSA | 39.8 | UJ |
| MW10A-GW-273-283-01-071824 | L41704-42 | d7-NMe-FOSE | 42.6 | UJ |
| MW10A-GW-273-283-01-071824 | L41704-42 | d9-NEt-FOSE | 41.7 | UJ |
| MW22B-GW-290-300-01-071824 | L41704-43 | 13C2-PFTeDA | 42.4 | UJ<br>PFTrDA: UJ |
| MW22B-GW-290-300-01-071824 | L41704-43 | D3-N-MeFOSA | 31.1 | UJ |
| MW22B-GW-290-300-01-071824 | L41704-43 | D5-N-EtFOSA | 24.4 | UJ |
| MW22B-GW-290-300-01-071824 | L41704-43 | d7-NMe-FOSE | 38.7 | UJ |
| MW22B-GW-290-300-01-071824 | L41704-43 | d9-NEt-FOSE | 36.8 | UJ |
| MW22A-GW-429-439-01-071824 | L41704-44 | 13C2-PFTeDA | 43.0 | UJ<br>PFTrDA: UJ |
| MW22A-GW-429-439-01-071824 | L41704-44 | D3-N-MeFOSA | 37.9 | UJ |
| MW22A-GW-429-439-01-071824 | L41704-44 | D5-N-EtFOSA | 26.2 | UJ |
| MW22A-GW-429-439-01-071824 | L41704-44 | d7-NMe-FOSE | 35.4 | UJ |
| MW22A-GW-429-439-01-071824 | L41704-44 | d9-NEt-FOSE | 31.7 | UJ |
| MW26B-GW-249-259-01-071824 | L41704-45 | 13C4-PFBA | 11.2 | J+ |
| MW26B-GW-249-259-01-071824 | L41704-45 | d9-NEt-FOSE | 45.6 | UJ |
| MW22C-GW-160-170-01-071824 | L41704-46 | 13C4-PFBA | 9.55 | R |
| MW22C-GW-160-170-01-071824 | L41704-46 | D5-N-EtFOSA | 46.2 | UJ |
| MW22C-GW-160-170-01-071824 | L41704-46 | d7-NMe-FOSE | 47.0 | UJ |
| MW22C-GW-160-170-01-071824 | L41704-46 | d9-NEt-FOSE | 43.7 | UJ |
| MW23A-GW-422-432-01-071824 | L41704-47 | 13C2-PFTeDA | 31.6 | UJ<br>PFTrDA: UJ |
| MW23A-GW-422-432-01-071824 | L41704-47 | D3-N-MeFOSA | 47.6 | UJ |
| MW23A-GW-422-432-01-071824 | L41704-47 | D5-N-EtFOSA | 35.8 | UJ |
| MW23A-GW-422-432-01-071824 | L41704-47 | d7-NMe-FOSE | 35.3 | UJ |
| MW23A-GW-422-432-01-071824 | L41704-47 | d9-NEt-FOSE | 28.9 | UJ |
| MW10B-GW-130-140-01-071824 | L41704-48 | 13C4-PFBA | 4.16 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW10B-GW-130-140-01-071824 | L41704-48 | 13C2-PFTeDA | 48.8 | UJ<br>PFTrDA: UJ |
| TRIP-Cooler1-072424 | L41740-1 | D3-N-MeFOSA | 30.7 | UJ |
| TRIP-Cooler1-072424 | L41740-1 | D5-N-EtFOSA | 26.3 | UJ |
| OW7S-3-GW-100-110-01-072424 | L41740-2 | 13C2-PFTeDA | 34.0 | UJ<br>PFTrDA: UJ |
| OW7S-3-GW-100-110-01-072424 | L41740-2 | D3-N-MeFOSA | 39.7 | UJ |
| OW7S-3-GW-100-110-01-072424 | L41740-2 | D5-N-EtFOSA | 31.4 | UJ |
| OW7S-3-GW-100-110-01-072424 | L41740-2 | d7-NMe-FOSE | 37.7 | UJ |
| OW7S-3-GW-100-110-01-072424 | L41740-2 | d9-NEt-FOSE | 30.7 | UJ |
| WC6A-GW-278-288-01-081524 | L41852-6 | D3-N-MeFOSA | 35.9 | UJ |
| WC6A-GW-278-288-01-081524 | L41852-6 | D5-N-EtFOSA | 27.4 | UJ |
| WC6A-GW-278-288-01-081524 | L41852-6 | d7-NMe-FOSE | 44.4 | UJ |
| WC6A-GW-278-288-01-081524 | L41852-6 | d9-NEt-FOSE | 46.2 | UJ |
| WC5A-GW-268-278-01-081524 | L41852-7 | 13C2-PFTeDA | 44.7 | UJ<br>PFTrDA: UJ |
| WC5A-GW-268-278-01-081524 | L41852-7 | D3-N-MeFOSA | 33.9 | UJ |
| WC5A-GW-268-278-01-081524 | L41852-7 | D5-N-EtFOSA | 25.4 | UJ |
| WC5A-GW-268-278-01-081524 | L41852-7 | d7-NMe-FOSE | 38.2 | UJ |
| WC5A-GW-268-278-01-081524 | L41852-7 | d9-NEt-FOSE | 35.9 | UJ |
| MW28A-GW-235-245-01-081524 | L41852-8 | 13C2-PFTeDA | 49.7 | UJ<br>PFTrDA: UJ |
| MW28A-GW-235-245-01-081524 | L41852-8 | D3-N-MeFOSA | 41.7 | UJ |
| MW28A-GW-235-245-01-081524 | L41852-8 | D5-N-EtFOSA | 33.5 | UJ |
| MW28A-GW-235-245-01-081524 | L41852-8 | d7-NMe-FOSE | 37.5 | UJ |
| MW28A-GW-235-245-01-081524 | L41852-8 | d9-NEt-FOSE | 32.5 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCS ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG90985-102/ WG90985-103 | PFDoS | **56.2/57.2** | UJ |
| | 11Cl-PF3OUdS | **64.5/66.4** | UJ |
| | 3:3 FTCA | **139/136** | No qualifier |
| | NFDHA | 112/**133** | No qualifier |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| PZED-GW-16-21-03-071824 | PFOA* | **138/229** | No qualifier |
| | PFHpS | **177/158** | J+ |
| | PFOS* | **1250/1290** | No qualifier |
| | PFNS | **44.9/66.1** | UJ |
| | PFDoS | **ND/17.1** | R |
| | PFDS | **9.41/43.5** | R |
| | N-EtFOSA | **137/120** | No qualifier |
| | 9Cl-PF3ONS | **28.0/55.0** | UJ |
| | 11Cl-PF3OUdS | **ND/26.0** | R |
| | 3:3 FTCA | **164/153** | No qualifier |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

ND – Not detected

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZED-GW-16-21-01-071824 | L41704-24 | FtS 6:2 ion |
| MW10A-GW-273-283-01-071824 | L41704-42 | PFOS |
| MW10B-GW-130-140-01-071824 | L41704-48 | PFNA |
| MW22A-GW-429-439-01-071824 | L41704-44 | FtS 6:2 ion |
| MW22B-GW-290-300-01-071824 | L41704-43 | PFDA |
| MW22C-GW-160-170-01-071824 | L41704-46 | PFDA |
| MW26B-GW-249-259-01-071824 | L41704-45 | PFOS |
| MW26C-GW-130-140-01-071824 | L41704-41 | PFOS |
| WC5A-GW-268-278-01-081524 | L41852-7 | PFOS |
| MW28A-GW-235-245-01-081524 | L41852-8 | PFOS |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG91385 (WG90985) Report date (mm/dd/yyyy): 9/20/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 3/19/2026

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples PZED-GW-16-21-01-071824, PZED-GW-16-21-02-071824, PZED-GW-16-21-03-071824 receipt templerature (11.8°C), TRIP-Cooler1-072424, OW7S-3-GW-100-110-01-072424 receipt temperature (9.8°C) were received above the recommended temperature limit (>6°C). Results for sample received at temperatures greater that 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used. ICAL and CCVs reviewed.<br>See data qualifications summary (attachment) for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-Cooler1-072424 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications.Results for two compounds are rejected (R) due to severely low recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | PZED-GW-16-21-03-071824 |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCS Low Level |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☒ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD<br>Field Triplicate: |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | PZED-GW-16-21-01-071824/PZED-GW-16-21-02-071824/PZED-GW-16-21-03-071824 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used for two samples. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (August 2024).**

**See attachment for additional comments and qualifications.**

**Results for two compounds are rejected (R) due to severely low surrogate recoveries, and nine results were rejected (R) due to severely low MS recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Priya Nagwanshi Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 3/19/2026

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93120 (WG92499)**

**Chain of custody preservation, and holding time (Section 1E):** Samples were analyzed 9 to 11 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were non-detects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below.One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Field Blank-101424 | L42263-1 | D5-NEtFOSA | 38.3 | UJ |
| Field Blank-101424 | L42263-1 | D3-NMeFOSA | 36.0 | UJ |
| Trip-Cooler1-101524 | L42263-2 | D9-NEtFOSE | 48.3 | UJ |
| Trip-Cooler1-101524 | L42263-2 | D7-NMeFOSE | 48.6 | UJ |
| Trip-Cooler1-101524 | L42263-2 | D5-NEtFOSA | 28.8 | UJ |
| Trip-Cooler1-101524 | L42263-2 | D3-NMeFOSA | 32.3 | UJ |
| Trip-Cooler2-101524 | L42263-3 | D5-NEtFOSA | 39.8 | UJ |
| Trip-Cooler2-101524 | L42263-3 | D3-NMeFOSA | 40.3 | UJ |
| Trip-Cooler3-101524 | L42263-4 | D5-NEtFOSA | 38.7 | UJ |
| Trip-Cooler3-101524 | L42263-4 | D3-NMeFOSA | 38.5 | UJ |
| Trip-Cooler4-101524 | L42263-5 | D5-NEtFOSA | 48.2 | UJ |
| Trip-Cooler4-101524 | L42263-5 | D3-NMeFOSA | 48.4 | UJ |
| RC5-WAT-BULK-01-101424 | L42263-6 | 13C2-PFTeDA | 36.9 | UJ<br>PFTrDA: UJ |
| RC5-WAT-BULK-01-101424 | L42263-6 | D5-NEtFOSA | 48.6 | UJ |
| RC5-WAT-BULK-02-101424 | L42263-7 | 13C2-PFTeDA | 34.4 | UJ<br>PFTrDA: UJ |
| RC5-WAT-BULK-02-101424 | L42263-7 | D5-NEtFOSA | 42.7 | UJ |
| RC5-WAT-BULK-02-101424 | L42263-7 | D9-NEtFOSE | 49.1 | UJ |
| RC5-WAT-BULK-03-101424 | L42263-8 (A) | 13C2-PFTeDA | 34.6 | UJ<br>PFTrDA: UJ |
| RC5-WAT-BULK-03-101424 | L42263-8 (A) | 13C2-4:2 FTS | 159 | No Qualifier |
| RC5-WAT-BULK-03-101424 | L42263-8 (A) | D5-NEtFOSA | 45.2 | UJ |
| OD5-WAT-BULK-01-101424 | L42263-9 | 13C2-PFTeDA | 31.8 | UJ<br>PFTrDA: UJ |
| OD5-WAT-BULK-01-101424 | L42263-9 | 13C2-4:2 FTS | 179 | No Qualifier |
| OD5-WAT-BULK-01-101424 | L42263-9 | D5-NEtFOSA | 41.9 | UJ |
| OD5-WAT-BULK-01-101424 | L42263-9 | D7-NMeFOSE | 49.3 | UJ |
| OD5-WAT-BULK-01-101424 | L42263-9 | D9-NEtFOSE | 41.5 | UJ |
| OD4-WAT-BULK-01-101424 | L42263-10 | 13C2-PFTeDA | 36.3 | UJ<br>PFTrDA: UJ |
| OD4-WAT-BULK-01-101424 | L42263-10 | D3-NMeFOSA | 49.9 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD4-WAT-BULK-01-101424 | L42263-10 | D5-NEtFOSA | 45.2 | UJ |
| OD4-WAT-BULK-01-101424 | L42263-10 | D9-NEtFOSE | 48.4 | UJ |
| OD3-WAT-BULK-01-101424 | L42263-11 | 13C2-PFTeDA | 34.4 | UJ<br>PFTrDA: UJ |
| OD3-WAT-BULK-01-101424 | L42263-11 | 13C2-4:2 FTS | 193 | No Qualifier |
| RC3-WAT-BULK-01-101424 | L42263-12 | 13C4-PFBA | 3.34 | R |
| RC3-WAT-BULK-01-101424 | L42263-12 | 13C5-PFPeA | 14.5 | UJ |
| RC3-WAT-BULK-01-101424 | L42263-12 | 13C2-PFTeDA | 42.6 | UJ<br>PFTrDA: UJ |
| RC3-WAT-BULK-01-101424 | L42263-12 | 13C2-4:2 FTS | 22.8 | UJ |
| RC3-WAT-BULK-01-101424 | L42263-12 | 13C3-HFPO-DA | 37.5 | UJ |
| RC23-WAT-BULK-01-101424 | L42263-13 | 13C2-PFTeDA | 31.9 | UJ<br>PFTrDA: UJ |
| RC23-WAT-BULK-01-101424 | L42263-13 | 13C2-4:2 FTS | 257 | No Qualifier |
| RC23-WAT-BULK-01-101424 | L42263-13 | D5-NEtFOSA | 47.0 | UJ |
| RC23-WAT-BULK-01-101424 | L42263-13 | D9-NEtFOSE | 48.6 | UJ |
| EQ-PERI-101424 | L42263-14 | D5-NEtFOSA | 42.1 | UJ |
| EQ-PERI-101424 | L42263-14 | D3-NMeFOSA | 43.5 | UJ |
| EQ-GEOSUB-101424 | L42263-15 | D9-NEtFOSE | 45.6 | UJ |
| EQ-GEOSUB-101424 | L42263-15 | D7-NMeFOSE | 46.1 | UJ |
| EQ-GEOSUB-101424 | L42263-15 | D5-NEtFOSA | 27.3 | UJ |
| EQ-GEOSUB-101424 | L42263-15 | D3-NMeFOSA | 27.6 | UJ |
| EQ-GEOSUB-101424 | L42263-15 | 13C2-PFTeDA | 45.3 | UJ<br>PFTrDA: UJ |
| SP42-GW-116-126-01-101424 | L42263-16 | 13C2-PFTeDA | 41.3 | UJ<br>PFTrDA: UJ |
| SP42-GW-116-126-01-101424 | L42263-16 | 13C2-4:2 FTS | 176 | No Qualifier |
| SP42-GW-116-126-01-101424 | L42263-16 | D3-NMeFOSA | 48.5 | UJ |
| SP42-GW-116-126-01-101424 | L42263-16 | D5-NEtFOSA | 45.0 | UJ |
| SP42-GW-116-126-01-101424 | L42263-16 | D3-NMeFOSAA | 134 | No Qualifier |
| SP42-GW-116-126-01-101424 | L42263-16 | D5-NEtFOSAA | 148 | J+ |
| SP42-GW-116-126-01-101424 | L42263-16 | D9-NEtFOSE | 35.9 | UJ |
| EQ-DIPPER-101424 | L42263-20 | D5-NEtFOSA | 29.3 | UJ |
| EQ-DIPPER-101424 | L42263-20 | D3-NMeFOSA | 32.2 | UJ |
| EQ-HYDRO-101424 | L42263-23 | D5-NEtFOSA | 37.7 | UJ |
| EQ-HYDRO-101424 | L42263-23 | D3-NMeFOSA | 38.4 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LSC/LCSD noncompliances are summarized below.

| LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG92499-102/ | PFDoS | **67.4/53.1** | UJ |

| LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG92499-103 | 11Cl-PF3OUdS | 82.2/**64.7** | UJ |
| | 7:3 FTCA | 71.9/**69.6** | UJ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| RC5-WAT-BULK-03-101424 | PFBA | **259/358** | J+ |
| | PFHxA | **202/153** | J+ |
| | PFHpA | 123/**143** | J+ |
| | PFOA | **481/776** | J+ |
| | PFTrDA | **67.5**/90.3 | UJ |
| | PFPeS | **143/137** | J+ |
| | PFHxS | **156/187** | J+ |
| | PFHpS | 125/**145** | J+ |
| | PFOS | **1270/3530** | J+ |
| | PFDoS | **46.9/56.1** | UJ |
| | PFOSA | 113/**141** | J+ |
| | 11Cl-PF3OUdS | **58.3/64.7** | UJ |

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OD4-WAT-BULK-01-101424 | L42263-10 | PFNA |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93200 (WG92500)**

**Chain of custody preservation, and holding time (Section 1G):** Samples were analyzed 15 to 25 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-).

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 01/28/25 16:49 | 3:3 FTCA | 136% | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV 01/28/25 19:55 | 3:3 FTCA | 138% | 70-130% | |
| CCV 01/29/25 00:35 | 3:3 FTCA | 133% | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 02/07/25 16:18 | 3:3 FTCA | 135% | 70-130% | |
| CCV 02/07/25 17:52 | 3:3 FTCA | 132% | 70-130% | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| PL41-GW-72-82-01-101424 | L42263-17 | 13C2-PFTeDA | 33.3 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-01-101424 | L42263-17 | D3-NMeFOSA | 14.3 | UJ |
| PL41-GW-72-82-01-101424 | L42263-17 | D5-NEtFOSA | 37.8 | UJ |
| PL41-GW-72-82-01-101424 | L42263-17 | D7-NMeFOSE | 40.5 | UJ |
| PL41-GW-72-82-01-101424 | L42263-17 | D9-NEtFOSE | 37.4 | UJ |
| PL41-GW-72-82-02-101424 | L42263-18 | 13C2-PFTeDA | 34.1 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-02-101424 | L42263-18 | D3-NMeFOSA | 43.2 | UJ |
| PL41-GW-72-82-02-101424 | L42263-18 | D5-NEtFOSA | 39.1 | UJ |
| PL41-GW-72-82-02-101424 | L42263-18 | D7-NMeFOSE | 43.0 | UJ |
| PL41-GW-72-82-02-101424 | L42263-18 | D9-NEtFOSE | 40.9 | UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | 13C2-PFDoA | 48.6 | UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | 13C2-PFTeDA | 25.8 | UJ<br>PFTrDA: UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | D3-NMeFOSA | 38.1 | UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | D5-NEtFOSA | 34.3 | UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | D7-NMeFOSE | 37.2 | UJ |
| PL41-GW-72-82-03-101424 | L42263-19 (A) | D9-NEtFOSE | 33.0 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BL5-WAT-BULK-01-101424 | L42263-21 | 13C2-PFTeDA | 41.5 | UJ<br>PFTrDA: UJ |
| BL5-WAT-BULK-01-101424 | L42263-21 | D5-NEtFOSA | 41.7 | UJ |
| BL5-WAT-BULK-01-101424 | L42263-21 | D7-NMeFOSE | 46.0 | UJ |
| BL5-WAT-BULK-01-101424 | L42263-21 | D9-NEtFOSE | 43.7 | UJ |
| BL5-WAT-BULK-02-101424 | L42263-22 | 13C2-PFTeDA | 32.2 | UJ<br>PFTrDA: UJ |
| BL5-WAT-BULK-02-101424 | L42263-22 | D3-NMeFOSA | 47.3 | UJ |
| BL5-WAT-BULK-02-101424 | L42263-22 | D5-NEtFOSA | 38.1 | UJ |
| BL5-WAT-BULK-02-101424 | L42263-22 | D7-NMeFOSE | 43.3 | UJ |
| BL5-WAT-BULK-02-101424 | L42263-22 | D9-NEtFOSE | 38.9 | UJ |
| RC2-WAT-BULK-01-101524 | L42263-24 | 13C2-PFTeDA | 34.9 | UJ<br>PFTrDA: UJ |
| RC2-WAT-BULK-01-101524 | L42263-24 | D7-NMeFOSE | 45.9 | UJ |
| RC2-WAT-BULK-01-101524 | L42263-24 | D9-NEtFOSE | 42.0 | UJ |
| RC2-WAT-BULK-02-101524 | L42263-25 | 13C2-PFTeDA | 37.9 | UJ<br>PFTrDA: UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | 13C2-PFDoA | 38.4 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | 13C2-PFTeDA | 14.5 | UJ<br>PFTrDA: UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D3-NMeFOSA | 44.3 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D5-NEtFOSA | 37.9 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D3-NMeFOSAA | 48.5 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D5-NEtFOSAA | 44.4 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D7-NMeFOSE | 31.5 | UJ |
| WL11-WAT-BULK-01-101524 | L42263-26 | D9-NEtFOSE | 27.1 | UJ |
| EP26D-WAT-BULK-01-101524 | L42263-27 | 13C2-PFTeDA | 37.6 | UJ<br>PFTrDA: UJ |
| EP26D-WAT-BULK-01-101524 | L42263-27 | D7-NMeFOSE | 44.5 | UJ |
| EP26D-WAT-BULK-01-101524 | L42263-27 | D9-NEtFOSE | 41.1 | UJ |
| WL6-WAT-BULK-01-101524 | L42263-28 | 13C2-PFTeDA | 29.8 | UJ<br>PFTrDA: UJ |
| WL6-WAT-BULK-01-101524 | L42263-28 | D7-NMeFOSE | 41.1 | UJ |
| WL6-WAT-BULK-01-101524 | L42263-28 | D9-NEtFOSE | 34.3 | UJ |
| EP16-WAT-BULK-01-101524 | L42263-29 | 13C2-PFTeDA | 41.4 | UJ<br>PFTrDA: UJ |
| WC3-WAT-BULK-01-101524 | L42263-30 | 13C2-PFTeDA | 38.8 | UJ<br>PFTrDA: UJ |
| WC3-WAT-BULK-01-101524 | L42263-30 | D5-NEtFOSA | 46.1 | UJ |
| WC5-WAT-BULK-01-101524 | L42263-31 | 13C2-PFTeDA | 30.2 | UJ<br>PFTrDA: UJ |
| WC5-WAT-BULK-01-101524 | L42263-31 | D5-NEtFOSA | 44.8 | UJ |
| WC5-WAT-BULK-01-101524 | L42263-31 | D7-NMeFOSE | 36.5 | UJ |
| WC5-WAT-BULK-01-101524 | L42263-31 | D9-NEtFOSE | 31.9 | UJ |
| WC4-WAT-BULK-01-101524 | L42263-32 | 13C2-PFTeDA | 32.7 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | | | PFTrDA: UJ |
| WC4-WAT-BULK-01-101524 | L42263-32 | D3-NMeFOSA | 43.8 | UJ |
| WC4-WAT-BULK-01-101524 | L42263-32 | D5-NEtFOSA | 37.0 | UJ |
| WC4-WAT-BULK-01-101524 | L42263-32 | D7-NMeFOSE | 45.8 | UJ |
| WC4-WAT-BULK-01-101524 | L42263-32 | D9-NEtFOSE | 42.1 | UJ |
| WC2-WAT-BULK-01-101524 | L42263-33 | 13C4-PFBA | 34.5 | J+ |
| WC2-WAT-BULK-01-101524 | L42263-33 | 13C2-PFTeDA | 35.8 | UJ<br>PFTrDA: UJ |
| WC2-WAT-BULK-01-101524 | L42263-33 | D9-NEtFOSE | 46.4 | UJ |
| WC6-WAT-BULK-01-101524 | L42263-34 | 13C4-PFBA | 30.0 | J+ |
| WC6-WAT-BULK-01-101524 | L42263-34 | 13C2-PFTeDA | 27.3 | UJ<br>PFTrDA: UJ |
| WC6-WAT-BULK-01-101524 | L42263-34 | D7-NMeFOSE | 46.8 | UJ |
| WC6-WAT-BULK-01-101524 | L42263-34 | D9-NEtFOSE | 41.2 | UJ |
| WC1-WAT-BULK-01-101524 | L42263-35 | 13C4-PFBA | 43.3 | J+ |
| WC1-WAT-BULK-01-101524 | L42263-35 | 13C2-PFTeDA | 36.6 | UJ<br>PFTrDA: UJ |
| WC1-WAT-BULK-01-101524 | L42263-35 | D9-NEtFOSE | 46.5 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LCS noncompliances are summarized below.

| LCS ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG92500-102/ WG92500-103 | PFDS | 85.5/**68.8** | UJ |
| | PFDoS | **69.6/45.2** | UJ |
| | 11Cl-PF3OUdS | **69.3/52.8** | UJ |
| | 3:3 FTCA | **134**/121 | No Qualifier |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | RPD | RPD Limit | Qualification of Associated Analyte |
|---|---|---|---|---|---|
| PL41-GW-72-82-03-101424 | PFBA | **259/362*** | 2.47 | 30 | No Qualifier |
| | PFHxA | 107/**226*** | 5.56 | 30 | No Qualifier |
| | PFOA | **158/196*** | 3.05 | 30 | No Qualifier |
| | PFDS | **71.5/54.5** | 27.2 | 30 | UJ |
| | PFDoS | **50.9/30.4** | **50.6** | 30 | UJ |
| | 11Cl-PF3OUdS | **51.1/36.8** | **32.7** | 30 | UJ |
| | 3:3 FTCA | **137/132** | 4.21 | 30 | No Qualifier |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| RC2-WAT-BULK-01-101524 | L42263-24 | PFOS |
| EP16-WAT-BULK-01-101524 | L42263-29 | PFDA |

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93160 (WG92501)**

**Chain of custody, preservation and holding time (Section 1.C and G):**

Samples were received at temperatures ranging from 7.8ºC to 9.6ºC, and above the limit of 6°C. Results were not qualified as the receipt temperatures were <10 ºC.

Samples were analyzed 8 to 23 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-). The sample collection date did not match the information between the chain-of-custody (COC) and the sample label for the sample LEPR-1-GW-100-140-01-101724. The collection date was listed as 17th October of 2024, while on the COC it is listed as 16th October of 2024. No further action is required.

**Calibration (Section 2):** Calibration noncompliance's are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 1/27/25 17:13 | 3:3 FTCA | **147** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 1/27/25 20:46 | 3:3 FTCA | **152** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 1/27/25 22:33 | 3:3 FTCA | **149** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 1/28/25 01:13 | 3:3 FTCA | **148** | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 2/05/25 18:16 | 11Cl-PF3OUdS | **137** | 70-130% | 11Cl-PF3OUdS results were not reported from these CCVs. No qualifiers. |
| CCV 2/05/25 18:55 | 11Cl-PF3OUdS | **142** | 70-130% | 11Cl-PF3OUdS results were not reported from these CCVs. No qualifiers. |
| CCV 2/06/25 16:36 | 11Cl-PF3OUdS | **134** | 70-130% | 11Cl-PF3OUdS results were not reported from these CCVs. No qualifiers. |

**Blanks-(Section 3Aii):** The analyte PFOA (0.480 J ng/L) was detected in method blank (WG92501-101). Results within 10 times the blank concentration were qualified BJ.

**Laboratory control spike/Laboratory control spike duplicates (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ) for the non-detects. Results associated with high recoveries were non-detects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG92501-102 (A/)/103 | PFDoS | **61.4/52.2** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG92501-102 (A)/103 | 11Cl-PF3OUdS | **63.2/55.4** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG92501-102 (A)/103 | 3:3 FTCA | **153/135** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG92501-102 (A/)/104 | PFDoS | **61.4/66.4** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG92501-102 (A/)/104 | 11Cl-PF3OUdS | **63.2/67.1** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG92501-102 (A/)/104 | 3:3 FTCA | **153/150** | 70-130% | All the associated sample results were not detected. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-Cooler 1-101724 | L42299-1 | D3-NMeFOSA | 34.3 | UJ |
| TRIP-Cooler 1-101724 | L42299-1 | D5-NEtFOSA | 29.7 | UJ |
| TRIP-Cooler 1-101724 | L42299-1 | D7-NMeFOSE | 49.6 | UJ |
| TRIP-Cooler 1-101724 | L42299-1 | D9-NEtFOSE | 48.2 | UJ |
| TRIP-Cooler 2-101724 | L42299-2 | D3-NMeFOSA | 31.9 | UJ |
| TRIP-Cooler 2-101724 | L42299-2 | D5-NEtFOSA | 26.0 | UJ |
| TRIP-Cooler 2-101724 | L42299-2 | D7-NMeFOSE | 44.2 | UJ |
| TRIP-Cooler 2-101724 | L42299-2 | D9-NEtFOSE | 42.9 | UJ |
| TRIP-Cooler 3-101724 | L42299-3 | D3-NMeFOSA | 30.0 | UJ |
| TRIP-Cooler 3-101724 | L42299-3 | D5-NEtFOSA | 25.2 | UJ |
| TRIP-Cooler 3-101724 | L42299-3 | D7-NMeFOSE | 44.7 | UJ |
| TRIP-Cooler 3-101724 | L42299-3 | D9-NEtFOSE | 43.9 | UJ |
| TRIP-Cooler 4-102124 | L42299-4 | D3-NMeFOSA | 33.2 | UJ |
| TRIP-Cooler 4-102124 | L42299-4 | D5-NEtFOSA | 29.7 | UJ |
| TRIP-Cooler 4-102124 | L42299-4 | D7-NMeFOSE | 47.4 | UJ |
| TRIP-Cooler 4-102124 | L42299-4 | D9-NEtFOSE | 48.1 | UJ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | 13C4-PFBA | 44.3 | J+ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | 13C2-PFTeDA | 27.2 | UJ<br>PFTrDA: UJ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | D3-NMeFOSA | 48.3 | UJ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | D5-NEtFOSA | 37.9 | UJ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | D7-NMeFOSE | 27.6 | UJ |
| LEPR-1-GW-100-140-01-101724 | L42299-5 | D9-NEtFOSE | 17.1 | UJ |
| Z-Op-GW-145-185-01-101624 | L42299-6 | D3-NMeFOSA | 48.1 | UJ |
| Z-Op-GW-145-185-01-101624 | L42299-6 | D5-NEtFOSA | 42.4 | UJ |
| MW10A-GW-273-283-01-101624 | L42299-7 | 13C4-PFBA | 45.2 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW10A-GW-273-283-01-101624 | L42299-7 | D5-NEtFOSA | 44.0 | UJ |
| MW10A-GW-273-283-01-101624 | L42299-7 | D7-NMeFOSE | 48.6 | UJ |
| MW10A-GW-273-283-01-101624 | L42299-7 | D9-NEtFOSE | 28.2 | UJ |
| MW10B-GW-130-140-01-101624 | L42299-8 | 13C4-PFBA | 43.1 | J+ |
| MW10B-GW-130-140-01-101624 | L42299-8 | D5-NEtFOSA | 41.3 | UJ |
| MW10B-GW-130-140-01-101624 | L42299-8 | D7-NMeFOSE | 48.9 | UJ |
| MW10B-GW-130-140-01-101624 | L42299-8 | D9-NEtFOSE | 31.8 | UJ |
| SiteC-GW-331-341-01-101724 | L42299-9 | 13C2-PFTeDA | 47.3 | UJ<br>PFTrDA: UJ |
| SiteC-GW-331-341-01-101724 | L42299-9 | D5-NEtFOSA | 38.7 | UJ |
| SiteC-GW-331-341-01-101724 | L42299-9 | D7-NMeFOSE | 38.9 | UJ |
| SiteC-GW-331-341-01-101724 | L42299-9 | D9-NEtFOSE | 24.5 | UJ |
| MW23C-GW-151-161-01-101724 | L42299-10 | 13C2-PFTeDA | 49.6 | UJ<br>PFTrDA: UJ |
| MW23C-GW-151-161-01-101724 | L42299-10 | D5-NEtFOSA | 40.0 | UJ |
| MW23C-GW-151-161-01-101724 | L42299-10 | D7-NMeFOSE | 48.1 | UJ |
| MW23C-GW-151-161-01-101724 | L42299-10 | D9-NEtFOSE | 34.2 | UJ |
| MW23B-GW-299-309-01-101724 | L42299-11 | 13C2-PFTeDA | 44.0 | UJ<br>PFTrDA: UJ |
| MW23B-GW-299-309-01-101724 | L42299-11 | D3-NMeFOSA | 44.2 | UJ |
| MW23B-GW-299-309-01-101724 | L42299-11 | D5-NEtFOSA | 33.5 | UJ |
| MW23B-GW-299-309-01-101724 | L42299-11 | D7-NMeFOSE | 46.3 | UJ |
| MW23B-GW-299-309-01-101724 | L42299-11 | D9-NEtFOSE | 27.7 | UJ |
| MW22C-GW-160-170-01-101724 | L42299-12 | D3-NMeFOSA | 33.7 | UJ |
| MW22C-GW-160-170-01-101724 | L42299-12 | D5-NEtFOSA | 25.5 | UJ |
| MW22C-GW-160-170-01-101724 | L42299-12 | D7-NMeFOSE | 45.4 | UJ |
| MW22C-GW-160-170-01-101724 | L42299-12 | D9-NEtFOSE | 31.1 | UJ |
| MW22B-GW-290-300-01-101724 | L42299-13 | 13C2-PFTeDA | 39.1 | UJ<br>PFTrDA: UJ |
| MW22B-GW-290-300-01-101724 | L42299-13 | D3-NMeFOSA | 41.7 | UJ |
| MW22B-GW-290-300-01-101724 | L42299-13 | D5-NEtFOSA | 29.0 | UJ |
| MW22B-GW-290-300-01-101724 | L42299-13 | D7-NMeFOSE | 45.8 | UJ |
| MW22B-GW-290-300-01-101724 | L42299-13 | D9-NEtFOSE | 33.3 | UJ |
| SiteJ-GW-358-368-01-101724 | L42299-14 | 13C2-PFTeDA | 47.1 | UJ<br>PFTrDA: UJ |
| SiteJ-GW-358-368-01-101724 | L42299-14 | D3-NMeFOSA | 46.5 | UJ |
| SiteJ-GW-358-368-01-101724 | L42299-14 | D5-NEtFOSA | 34.7 | UJ |
| SiteJ-GW-358-368-01-101724 | L42299-14 | D7-NMeFOSE | 42.7 | UJ |
| SiteJ-GW-358-368-01-101724 | L42299-14 | D9-NEtFOSE | 24.9 | UJ |
| M_Op-GW-127-167-01-101624 | L42299-15 | 13C2-PFTeDA | 32.3 | UJ<br>PFTrDA: UJ |
| M_Op-GW-127-167-01-101624 | L42299-15 | D3-NMeFOSA | 46.4 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| M_Op-GW-127-167-01-101624 | L42299-15 | D5-NEtFOSA | 35.2 | UJ |
| M_Op-GW-127-167-01-101624 | L42299-15 | D7-NMeFOSE | 41.5 | UJ |
| M_Op-GW-127-167-01-101624 | L42299-15 | D9-NEtFOSE | 33.5 | UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | 13C2-PFTeDA | 44.3 | UJ<br>PFTrDA: UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | D3-NMeFOSA | 47.3 | UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | D5-NEtFOSA | 36.1 | UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | D5-NEtFOSAA | 49.0 | UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | D7-NMeFOSE | 40.1 | UJ |
| MW8B-GW-50-60-01-101624 | L42299-16 | D9-NEtFOSE | 38.3 | UJ |
| MW8B-GW-50-60-02-101624 | L42299-17 | 13C2-PFTeDA | 41.2 | UJ<br>PFTrDA: UJ |
| MW8B-GW-50-60-02-101624 | L42299-17 | D3-NMeFOSA | 35.4 | UJ |
| MW8B-GW-50-60-02-101624 | L42299-17 | D5-NEtFOSA | 28.4 | UJ |
| MW8B-GW-50-60-02-101624 | L42299-17 | D7-NMeFOSE | 38.4 | UJ |
| MW8B-GW-50-60-02-101624 | L42299-17 | D9-NEtFOSE | 36.4 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | 13C2-PFTeDA | 36.2 | UJ<br>PFTrDA: UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | 13C8-PFOSA | 42.0 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | D3-NMeFOSA | 27.4 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | D5-NEtFOSA | 22.6 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | D5-NEtFOSAA | 49.7 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | D7-NMeFOSE | 31.0 | UJ |
| W6201-GW-109-124-01-101624 | L42299-18 | D9-NEtFOSE | 26.4 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW10A-GW-273-283-01-101624 | L42299-7 | PFOS |
| MW10B-GW-130-140-01-101624 | L42299-8 | PFOS |
| SiteJ-GW-358-368-01-101724 | L42299-14 | PFOS |
| M_Op-GW-127-167-01-101624 | L42299-15 | PFOS |
| W6201-GW-109-124-01-101624 | L42299-18 | PFOS |

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93287 (WG92502)**

**Chain of custody, preservation and holding time (Section 1 C and F):**

Samples were received at temperatures ranging from 7.8ºC to 9.6ºC, and above the limit of 6°C. Results were not qualified as the receipt temperatures were <10 ºC

Samples were analyzed 19 to 27 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-).

**Calibration (Section 2):** Calibration noncompliance's are summarized below.

| Calibration | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| CCV 2/03/25 17:12 | 3:3 FTCA | **178** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 17:12 | 7:3 FTCA | **138** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 17:12 | PFEESA | **131** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 20:32 | 3:3 FTCA | **168** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 20:32 | 7:3 FTCA | **135** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 22:45 | 3:3 FTCA | **168** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 22:45 | 7:3 FTCA | **139** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/03/25 22:45 | PFEESA | **131** | 70-130% | The associated sample results were not detected. Hence data qualification was not considered necessary. No qualifiers. |
| CCV 2/04/25 01:12 | 3:3 FTCA | **171** | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 2/04/25 01:12 | 7:3 FTCA | **136** | 70-130% | 7:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 2/04/25 01:12 | PFEESA | **131** | 70-130% | PFEESA results were not reported from these CCVs. No qualifiers. |
| CCV 2/11/25 15:06 | 3:3 FTCA | **140** | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |
| CCV 2/11/25 17:33 | 3:3 FTCA | **140** | 70-130% | 3:3 FTCA results were not reported from these CCVs. No qualifiers. |

**Blanks (Section 3Aii):**

The analyte PFOA (0.519 J ng/L) was detected in the method blank (WG92502-101). Results within 10 times the blank concentration were qualified BJ.

**Laboratory control spike/Laboratory control spike duplicates – (Section 5Bii):** Results associated with low recoveries were qualified as estimated (UJ) for the non-detects. Results associated with high recoveries were non-detects and were acceptable without qualification.

| LCS/LCSD ID | Analyte | %R | %R Limits | Qualifiers |
|---|---|---|---|---|
| WG92502-102 /104 | PFDoS | **66.6/67.7** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |
| WG92502-103 | PFDoS | **68.0** | 70-130% | |
| WG92502-102 (A)/104 | 3:3 FTCA | **156/180** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG92502-103 | 3:3 FTCA | **155** | 70-130% | |
| WG92502-102 (A)/104 | 7:3 FTCA | **128/135** | 70-130% | All the associated sample results were not detected. No qualifiers. |
| WG92502-103 | 7:3 FTCA | **134** | 70-130% | |
| WG92502-103 | 11Cl-PF3OUdS | **67.5** | 70-130% | Results associated with low recoveries were qualified and estimated as UJ. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| W6102-GW-93-105-01-101624 | L42299-19 | D5-NEtFOSA | 49.0 | UJ |
| W6102-GW-93-105-01-101624 | L42299-19 | D9-NEtFOSE | 48.1 | UJ |
| WC-6A-GW-278-288-01-101624 | L42299-24 | 13C4-PFBA | 36.4 | J+ |
| WC-6A-GW-278-288-01-101624 | L42299-24 | D9-NEtFOSE | 38.7 | UJ |
| MW25A-GW-390-400-01-101724 | L42299-25 | 13C4-PFBA | 22.9 | J+ |
| MW25B-GW-260-270-01-101724 | L42299-26 | 13C4-PFBA | 14.8 | J+ |
| MW25B-GW-260-270-01-101724 | L42299-26 | 13C2-8:2 FTS | 46.7 | UJ |
| MW25B-GW-260-270-01-101724 | L42299-26 | D9-NEtFOSE | 36.1 | UJ |
| OW7S-1-GW-90-100-01-101724 | L42299-30 | D5-NEtFOSAA | 44.0 | UJ |
| WC5A-GW-268-278-01-101724 | L42299-32 | 13C4-PFBA | 32.3 | J+ |
| WC5A-GW-268-278-01-101724 | L42299-32 | D9-NEtFOSE | 35.4 | UJ |
| C_WT-GW-48-58-01-101724 | L42299-33 | 13C2-PFTeDA | 25.1 | UJ<br>PFTrDA: UJ |
| C_WT-GW-48-58-01-101724 | L42299-33 | D5-NEtFOSA | 40.1 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| C_WT-GW-48-58-01-101724 | L42299-33 | D9-NEtFOSE | 47.1 | UJ |
| DD-GW-63-73-01-101724 | L42299-34 | 13C2-PFTeDA | 4.17 | R<br>PFTrDA: R |
| DD-GW-63-73-01-101724 | L42299-34 | D5-NEtFOSA | 37.1 | UJ |
| DD-GW-63-73-01-101724 | L42299-34 | D5-NEtFOSAA | 48.1 | UJ |
| DD-GW-63-73-01-101724 | L42299-34 | D7-NMeFOSE | 35.0 | UJ |
| DD-GW-63-73-01-101724 | L42299-34 | D9-NEtFOSE | 24.9 | UJ |
| DD-GW-63-73-01-101724 | L42299-34 | 13C2-PFDoA | 40.2 | UJ |
| MW28B-GW-119-129-01-101824 | L42299-35 | 13C4-PFBA | 26.2 | J+ |
| MW28B-GW-119-129-01-101824 | L42299-35 | D5-NEtFOSA | 49.5 | UJ |
| MW28B-GW-119-129-01-101824 | L42299-35 | D3-NMeFOSAA | 48.7 | UJ |
| PZH-1-GW-90-100-01-101824 | L42299-36 | 13C4-PFBA | 38.0 | J+ |
| PZH-1-GW-90-100-01-101824 | L42299-36 | D9-NEtFOSE | 44.2 | UJ |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW25B-GW-260-270-01-101724 | L42299-26 | PFOS |
| MW26B-GW-249-259-01-101724 | L42299-27 | PFOS |
| MW26C-GW-130-140-01-101724 | L42299-28 | PFOS |
| WC5A-GW-268-278-01-101724 | L42299-32 | PFOS |
| DD-GW-63-73-01-101724 | L42299-34 | PFOS |
| MW28B-GW-119-129-01-101824 | L42299-35 | PFOS |
| PZH-1-GW-90-100-01-101824 | L42299-36 | PFOS |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93049 (WG92503)**

**Chain of custody, preservation and holding time (Section 1.C and F):**

Samples were received at receipt temperatures ranging from 7.8°C to 15.4°C, and were above the criteria of 6°C. Results for samples received at temperatures greater that 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects (samples MW22A-GW-429-439-01-102124, MW28A-GW-235-245-01-102124, SiteI-GW-411-421-01-102124 and MW2D-GW-7-17-01-102124).

Samples were analyzed 3 to 12 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with high recoveries were nondetects and were acceptable without qualification. Labeled analog exceedances and qualifications are summarized in the table below. Two results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW20A-GW-130-140-01-101824 | L42299-37 | 13C4-PFBA | 12.3 | R |
| MW20A-GW-130-140-01-101824 | L42299-37 | 13C2-PFTeDA | 35.7 | UJ<br>PFTrDA: UJ |
| MW20A-GW-130-140-01-101824 | L42299-37 | D9-NEtFOSE | 39.5 | UJ |
| OW20S-1-GW-190-200-01-101824 | L42299-38 | 13C2-PFTeDA | 46.9 | UJ<br>PFTrDA: UJ |
| OW20S-1-GW-190-200-01-101824 | L42299-38 | D3-NMeFOSAA | 152 | No Qualifier |
| OW20S-1-GW-190-200-01-101824 | L42299-38 | D5-NEtFOSAA | 157 | No Qualifier |
| OW20S-1-GW-190-200-01-101824 | L42299-38 | D9-NEtFOSE | 46.1 | UJ |
| OW20P-1-GW-150-160-01-101824 | L42299-39 | 13C4-PFBA | 24.4 | J+ |
| OW20P-1-GW-150-160-01-101824 | L42299-39 | 13C2-PFTeDA | 44.6 | UJ<br>PFTrDA: UJ |
| OW20P-1-GW-150-160-01-101824 | L42299-39 | 13C2-4:2 FTS | 162 | No Qualifier |
| OW20P-1-GW-150-160-01-101824 | L42299-39 | D9-NEtFOSE | 43.3 | UJ |
| OW20T-1-GW-98-108-01-101824 | L42299-40 | 13C2-PFTeDA | 40.2 | UJ<br>PFTrDA: UJ |
| OW20T-1-GW-98-108-01-101824 | L42299-40 | D9-NEtFOSE | 45.3 | UJ |
| OW20S-3-GW-165-175-01-101824 | L42299-41 | 13C4-PFBA | 9.45 | R |
| OW20S-3-GW-165-175-01-101824 | L42299-41 | 13C2-4:2 FTS | 325 | No Qualifier |
| OW20S-3-GW-165-175-01-101824 | L42299-41 | D9-NEtFOSE | 46.6 | UJ |
| PZHD-GW-21-26-01-101824 | L42299-42 | 13C2-PFTeDA | 37.0 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | | | PFTrDA: UJ |
| PZHD-GW-21-26-01-101824 | L42299-42 | D3-NMeFOSA | 47.0 | UJ |
| PZHD-GW-21-26-01-101824 | L42299-42 | D5-NEtFOSA | 44.3 | UJ |
| PZGD-GW-35-40-01-101824 | L42299-43 | 13C2-PFTeDA | 35.2 | UJ<br>PFTrDA: UJ |
| PZGD-GW-35-40-01-101824 | L42299-43 | D3-NMeFOSA | 42.1 | UJ |
| PZGD-GW-35-40-01-101824 | L42299-43 | D5-NEtFOSA | 36.3 | UJ |
| PZGD-GW-35-40-01-101824 | L42299-43 | D9-NEtFOSE | 49.7 | UJ |
| PZGS-GW-20-30-01-101824 | L42299-44 | 13C2-PFTeDA | 27.7 | UJ<br>PFTrDA: UJ |
| PZGS-GW-20-30-01-101824 | L42299-44 | D3-NMeFOSA | 33.2 | UJ |
| PZGS-GW-20-30-01-101824 | L42299-44 | D5-NEtFOSA | 30.4 | UJ |
| PZGS-GW-20-30-01-101824 | L42299-44 | D7-NMeFOSE | 39.0 | UJ |
| PZGS-GW-20-30-01-101824 | L42299-44 | D9-NEtFOSE | 35.5 | UJ |
| TRIP-COOLER1-102324 | L42373-1 | D5-NEtFOSA | 27.7 | UJ |
| TRIP-COOLER1-102324 | L42373-1 | D3-NMeFOSA | 31.9 | UJ |
| TRIP-COOLER1-102324 | L42373-1 | 13C2-PFTeDA | 49.0 | UJ<br>PFTrDA: UJ |
| TRIP-COOLER2-102324 | L42373-2 | D5-NEtFOSA | 33.1 | UJ |
| TRIP-COOLER2-102324 | L42373-2 | D3-NMeFOSA | 34.2 | UJ |
| TRIP-COOLER3-102324 | L42373-3 | D9-NEtFOSE | 49.1 | UJ |
| TRIP-COOLER3-102324 | L42373-3 | D5-NEtFOSA | 29.7 | UJ |
| TRIP-COOLER3-102324 | L42373-3 | D3-NMeFOSA | 32.1 | UJ |
| TRIP-COOLER3-102324 | L42373-3 | 13C2-PFTeDA | 49.2 | UJ<br>PFTrDA: UJ |
| MW28A-GW-235-245-01-102124 | L42373-5 | 13C2-PFTeDA | 44.3 | UJ<br>PFTrDA: UJ |
| MW28A-GW-235-245-01-102124 | L42373-5 | D9-NEtFOSE | 47.7 | UJ |
| SiteI-GW-411-421-01-102124 | L42373-6 | 13C2-PFTeDA | 31.3 | UJ<br>PFTrDA: UJ |
| SiteI-GW-411-421-01-102124 | L42373-6 | D7-NMeFOSE | 49.8 | UJ |
| SiteI-GW-411-421-01-102124 | L42373-6 | D9-NEtFOSE | 38.2 | UJ |
| MW2D-GW-7-17-01-102124 | L42373-7 | 13C2-PFTeDA | 41.6 | UJ<br>PFTrDA: UJ |
| MW2D-GW-7-17-01-102124 | L42373-7 | D3-NMeFOSA | 43.4 | UJ |
| MW2D-GW-7-17-01-102124 | L42373-7 | D5-NEtFOSA | 38.4 | UJ |
| MW2D-GW-7-17-01-102124 | L42373-7 | D7-NMeFOSE | 49.7 | UJ |
| MW2D-GW-7-17-01-102124 | L42373-7 | D9-NEtFOSE | 46.3 | UJ |
| V-GW-63-71-01-101824 | L42373-12 | 13C2-PFDoA | 40.9 | UJ |
| V-GW-63-71-01-101824 | L42373-12 | 13C2-PFTeDA | 34.2 | UJ<br>PFTrDA: UJ |
| V-GW-63-71-01-101824 | L42373-12 | D3-NMeFOSA | 28.2 | UJ |
| V-GW-63-71-01-101824 | L42373-12 | D5-NEtFOSA | 28.8 | UJ |
| V-GW-63-71-01-101824 | L42373-12 | D7-NMeFOSE | 34.3 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| V-GW-63-71-01-101824 | L42373-12 | D9-NEtFOSE | 36.7 | UJ |
| V-GW-63-71-02-101824 | L42373-13 | 13C2-PFTeDA | 48.2 | UJ<br>PFTrDA: UJ |
| V-GW-63-71-02-101824 | L42373-13 | D3-NMeFOSA | 39.7 | UJ |
| V-GW-63-71-02-101824 | L42373-13 | D5-NEtFOSA | 40.2 | UJ |
| V-GW-63-71-02-101824 | L42373-13 | D7-NMeFOSE | 45.1 | UJ |
| V-GW-63-71-02-101824 | L42373-13 | D9-NEtFOSE | 48.2 | UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | 13C2-PFDoA | 46.5 | UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | 13C2-PFTeDA | 37.4 | UJ<br>PFTrDA: UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | D3-NMeFOSA | 39.4 | UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | D5-NEtFOSA | 33.5 | UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | D7-NMeFOSE | 38.8 | UJ |
| V-GW-63-71-03-101824 | L42373-14 (A) | D9-NEtFOSE | 42.6 | UJ |

**Laboratory control sample / Laboratory control sample duplicates (Section 5):** LCS noncompliances are summarized below.

| LCS/LCSD ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| WG92503-102/ WG92503-103 | PFDoS | **63.9/56.8** | UJ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| V-GW-63-71-03-101824 | PFBA | 94.9/**144*** | No Qualifier |
| | PFOA | **NR/67.7 *** | No Qualifier |
| | PFNS | **73.0/68.9** | UJ |
| | PFDS | **58.4/52.5** | UJ |
| | PFDoS | **47.2/52.6** | UJ |
| | 11Cl-PF3OUdS | **48.6/47.6** | UJ |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| MW2D-GW-7-17-01-102124 | L42373-7 | N-EtFOSAA |
| PZHD-GW-21-26-01-101824 | L42299-42 | N-EtFOSAA |
| OW20S-1-GW-190-200-01-101824 | L42299-38 | PFOS |
| OW20P-1-GW-150-160-01-101824 | L42299-39 | PFOS |

| Sample ID | Lab ID | Analyte |
|---|---|---|
| OW20S-3-GW-165-175-01-101824 | L42299-41 | PFOS |
| OW20T-1-GW-98-108-01-101824 | L42299-40 | PFOS |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG93042 (WG92504)**

**Chain of custody, preservation and holding time (Section 1.C and F):**

Samples were received at receipt temperatures of 15.3°C and 15.4°C, and were above the criteria of 6°C. Results for sample received at temperatures greater that 10°C were qualified as estimated (J-) for detects and (UJ) for non-detects (all samples).

Samples were analyzed 8 to 14 days over the 90-day SGS AXYS extended-time storage time limit and were qualified as estimated (UJ and J-).

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. One result was rejected (R) due to a severely low labeled analog recovery.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW26A-GW-380-390-01-102124 | L42373-8 | 13C4-PFBA | 3.65 | R |
| MW26A-GW-380-390-01-102124 | L42373-8 | 13C5-PFPeA | 27.3 | J+ |

**Matrix spike / Matrix spike duplicates (Section 6):** MS/MSD noncompliances are summarized below.

| MS/MSD sample ID | Analyte | % Recovery (limits 70% to 130%) | Qualification of Associated Analyte |
|---|---|---|---|
| PZED-GW-16-21-03-102324 | PFOA | 87.4/**13.9*** | No Qualifier |
| | PFOS | **195/336*** | No Qualifier |

*MS/MSD recovery limits did not apply (when the sample concentration is ≥ 4x the spike added, as indicated in National Functional Guidelines).

**Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios). These results were reported as maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| Q3-GW-110-126-01-101824 | L42373-19 | PFHpS |
| PZED-GW-16-21-01-102324 | L42373-21 | N-EtFOSAA |
| MW2F-GW-130-150-01-101824 | L42373-20 | N-EtFOSAA |
| MW26A-GW-380-390-01-102124 | L42373-8 | PFBA |

Results associated with both high and low bias were qualified as estimated (J), with no bias flag.

# Attachment H-2
# AXYS Laboratory Validation Packages (SAFF® Samples)

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86050 (WG85602)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ). Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-230320 | L39012-1 | 13C4-PFBA | 49.3 | UJ |
| Batch1-T0-230228 | L39012-10 | d7-NMe-FOSE | 151 | No Qualifier |
| Batch1-T125-230324 | L39083-2 | d7-NMe-FOSE | 152 | No Qualifier |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Not all compounds reported were included in the OPRs (LCSs). Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS / LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85602-102(A) / WG85602-103 | 4:2 FTS | 0 / 0 | 70 - 130 | Associated results were nondetect and were rejected (R) due to the severely low recoveries.<br><br>All samples were associated with this LCS/LCSD data. |
| | 6:2 FTS | 0.6 / 1.1 | 70 - 130 | |
| | 8:2 FTS | 1.3 / 0.9 | 70 - 130 | |
| | MeFOSAA | 0.1 / 0.2 | 70 - 130 | |
| | EtFOSAA | 0.1 / 0.1 | 70 - 130 | |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG86050 (WG85602) Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/17/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | TRIP-COOLER1-230320<br>TRIP-COOLER1-230324 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL:<br>6:2 FTS 19.6 ng/L |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | Not all compounds reported included in the OPRs (LCSs) |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. Results for five compounds are rejected (R) due to severely low recoveries. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD RPDs ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix.<br>LCS/LCSD RPDs ok (not all compounds reported) |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Reduced sample volumes used. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

**Results for five compounds (all samples) are rejected (R) due to severely low OPR (LCS/LCSD) recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 4/17/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86528 (WG85919)**

**Chain of custody preservation, and holding time (Section 1C):** Nine samples were received at temperatures greater than the 6 °C criteria at 6.4 and 7.0 °C. Results were not qualified, as the samples were received at temperatures < 10 °C.

A sample collection date and time were not listed on the chain of custody for sample SP2-02-EFF1-230303-R55 (L39031-30).

Samples were extracted 5 to 26 days over the 90-day extended hold time. Sample detects were qualified as estimated biased low (J-), and nondetects were qualified UJ.

**Trip blank and equipment blank detections (Section 3Aii):** Associated trip blank detections are listed in the table below (these trip blanks were not reported in this SDG). Associated sample detects within 10 times the blank concentrations were qualified BJ.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| TRIP-COOLER4-032223 | 6:2 FTS | 31.6 J | Associated results were nondetect. |
| | PFHxA | 4.47 J | Associated sample detects that were within 10 times the blank concentration were qualified BJ:<br><br>SP1-02-T1-230227-R51 (L39031-35)<br>SP2-02-EFF1-230227-R51 (L39031-36)<br>SP2-02-EFF1-230228-R52 (L39031-37) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were nondetect and qualified as estimated (UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP1-02-T1-230307-R56 | L39031-32 | d9-NEt-FOSE | 157 | UJ |
| SP1-02-T1-230227-R51 | L39031-35 | d9-NEt-FOSE | 167 | UJ |
| SP2-02-EFF1-230227-R51 | L39031-36 | d9-NEt-FOSE | 154 | UJ |
| SP1-02-T1-230313-R57 | L39031-38 | d9-NEt-FOSE | 166 | UJ |
| SP1-02-T1-230313-R57-DUP | L39031-39 | d9-NEt-FOSE | 151 | UJ |
| SP1-02-T1SEC-230320-R57 | L39031-48 | d9-NEt-FOSE | 160 | UJ |
| SP1-02-T1SEC-230320-R57R | L39031-50 | d9-NEt-FOSE | 159 | UJ |

**MS/MSD Exceedances (Section 6Av):** MS/MSD recoveries and relative percent differences (RPDs) were outside the acceptable limits as listed below.

| Compound | MS/MSD % Recovery | Recovery Limits | RPD | RPD Limit | Results Qualified |
|---|---|---|---|---|---|
| **SP1-02-T1-230313-R57-MS/MSD:** | | | | | |
| PFBA | **Not Reported / 189** | 70-130 | 20.7 | 30 | The sample result was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **200 / 276** | 70-130 | 2.08 | 30 | |
| PFOS | **634 / 649** | 70-130 | 0.031 | 30 | |

**Bold indicates an exceedance**

**Method detection limits/Report limits (Section 7B):**

Reduced sample volumes were used for samples SP3-01-T3-230306-R56 ((L39031-29), SP4-01-T4-230303-R55 (L39031-31), SP3-01-T3-230307-R56 (L39031-34), SP3-01-T3-230313-R57 (L39031-46), and SP4-01-T4-230320-R57 (L39031-47) due to high sample concentrations resulting in elevated method reporting limits.

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as maximum possible concentrations and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230303-R55 | L39031-30 | PFHpS |

PFOS results were reported from dilution analyses for samples SP1-02-T1-230307-R56 (L39031-32), SP1-02-T1-230227-R51 (L39031-35), SP1-02-T1-230313-R57 (L39031-38), SP1-02-T1-230313-R57-DUP (L39031-39), SP1-02-T1-230313-R57-MS/MSD (L39031-40), SP1-02-T1SEC-230320-R57 (L39031-48), and SP1-02-T1SEC-230320-R57R (L39031-50) due to high concentrations.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: DPWG86528 (WG85919) Report date (mm/dd/yyyy): 8/9/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/18/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Nine samples were received at temperatures of above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for holding time exceedances and qualifications. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature and holding exceedances are discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in the SDG:<br>TRIP-COOLER3-032223<br>TRIP-COOLER4-032223<br>TRIP-COOLER5-032223<br>TRIP-COOLER6-032223 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for a list of trip blank detections and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | SP1-02-T1-230313-R57-MS/MSD |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSD RPDs ok<br>Field Triplicate:<br>SP1-02-T1-230313-R57 /<br>SP1-02-T1-230313-R57-DUP /<br>SP1-02-T1-230313-R57-MS/MSD |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for details. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/18/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32203351**

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SAFF-Inlet-112222 | 32203351001 | PFBA |
| SAFF-Inlet-112222 | 32203351001 | PFOA |
| SAFF-Inlet-112222 | 32203351001 | PFOS |
| SAFF-Outlet-112222 | 32203351002 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32203351 Report date (mm/dd/yyyy): 11/30/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | LCSD RPD ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32203401**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The laboratory indicated that the labeled analog recoveries were low due to a lack of preservative. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP3-01-T3-221129-R1 15mL | 32203401008 | 13C4-PFBA | 25.0 | J+ |
| SP3-01-T3-221129-R1 15mL | 32203401008 | 13C5-PFPeA | 28.2 | J+ |
| SP3-01-T3-221129-R1 15mL | 32203401008 | 13C5-PFHxA | 35.3 | J+ |
| SP3-01-T3-221129-R1 15mL | 32203401008 | 13C4-PFHpA | 44.0 | J+ |
| SP3-01-T3-221129-R1 15mL | 32203401008 | 13C3-HFPO-DA | 27.4 | Not Reported |
| SP4-01-T4-221129-R1 | 32203401011 | 13C4-PFBA | 40.7 | J+ |
| SP4-01-T4-221129-R1 | 32203401011 | 13C5-PFPeA | 42.8 | J+ |
| SP4-01-T4-221129-R1 | 32203401011 | 13C5-PFHxA | 48.8 | J+ |
| SP4-01-T4-221129-R1 | 32203401011 | 13C3-HFPO-DA | 43.3 | Not Reported |

**10. Additional comments about lab report:** Thirty results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-221128-R2 | 32203401001 | PFBA |
| SP1-02-T1-221128-R2 | 32203401001 | PFOA |
| SP1-02-T1-221128-R2 | 32203401001 | PFOS |
| SP2-01-EFF1-221128-R2 | 32203401002 | PFBA |
| SP2-01-EFF1-221128-R2 | 32203401002 | PFOA |
| SP2-01-EFF1-221128-R2 | 32203401002 | PFOS |
| SP2-01-EFF1-221128-R3 | 32203401003 | PFBA |
| SP2-01-EFF1-221128-R3 | 32203401003 | PFOA |
| SP2-01-EFF1-221128-R3 | 32203401003 | PFOS |
| SP2-01-EFF1-221128-R4 | 32203401004 | PFBA |
| SP2-01-EFF1-221128-R4 | 32203401004 | PFOA |
| SP2-01-EFF1-221128-R4 | 32203401004 | PFOS |
| SP2-01-EFF1-221129-R1 | 32203401005 | PFBA |
| SP2-01-EFF1-221129-R1 | 32203401005 | PFOA |
| SP2-01-EFF1-221129-R1 | 32203401005 | PFOS |
| SP2-01-EFF1-221129-R2 | 32203401006 | PFBA |
| SP2-01-EFF1-221129-R2 | 32203401006 | PFOA |
| SP2-01-EFF1-221129-R2 | 32203401006 | PFOS |
| SP2-01-EFF1-221129-R3 | 32203401007 | PFBA |
| SP2-01-EFF1-221129-R3 | 32203401007 | PFOA |
| SP2-01-EFF1-221129-R3 | 32203401007 | PFOS |
| SP3-01-T3-221129-R1 15mL | 32203401008 | PFOS |

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-SEC-221129-R1 | 32203401010 | PFBA |
| SP1-02-T1-SEC-221129-R1 | 32203401010 | PFOA |
| SP1-02-T1-SEC-221129-R1 | 32203401010 | PFOS |
| SP4-01-T4-221129-R1 | 32203401011 | PFOA |
| SP4-01-T4-221129-R1 | 32203401011 | PFOS |
| SP2-01-EFF2-221129-R1 | 32203401012 | PFBA |
| SP2-01-EFF2-221129-R1 | 32203401012 | PFOA |
| SP2-01-EFF2-221129-R1 | 32203401012 | PFOS |

**10. Additional comments about lab report:** Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32203401 Report date (mm/dd/yyyy): 12/5/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | LCSD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples SP3-01-T3-221129-R1 15mL and SP4-01-T4-221129-R1 has elevated RLs due to samples arriving with lower sample size. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32203443**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C5-PFHxA | 49.5 | J+ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C4-PFHpA | 41.3 | J+ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C6-PFDA | 44.9 | J+ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C7-PFuNA | 48.4 | UJ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C8-PFOA | 40.2 | J+ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C9-PFNA | 43.8 | J+ |
| SP2-01-EFF1-221205-R1 | 32203443002 | 13C3-HFPO-DA | 47.4 | Not Reported |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C4-PFBA | 12.2 | J+ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C5-PFPeA | 15.6 | UJ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C5-PFHxA | 19.3 | J+ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C4-PFHpA | 26.2 | J+ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C8-PFOA | 36.9 | J+ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C9-PFNA | 49.7 | J+ |
| SP3-01-T3-221205-R1 15mL | 32203443003 | 13C3-HFPO-DA | 14.7 | Not Reported |

**10. Additional comments about lab report:** Seven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-221205-R1 | 32203443001 | PFBA |
| SP1-02-T1-221205-R1 | 32203443001 | PFOA |
| SP1-02-T1-221205-R1 | 32203443001 | PFOS |
| SP2-01-EFF1-221205-R1 | 32203443002 | PFBA |
| SP2-01-EFF1-221205-R1 | 32203443002 | PFOA |
| SP2-01-EFF1-221205-R1 | 32203443002 | PFOS |
| SP3-01-T3-221205-R1 15mL | 32203443003 | PFOS |

**10. Additional comments about lab report:** Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32203443 Report date (mm/dd/yyyy): 12/9/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | LCSD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP3-01-T3-221205-R1-15nL has elevated RLs due to the sample arriving with a lower sample size. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Sample SP3-01-T3-221205-R1 50mL was placed on hold and results were not reported.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32203536**

**10. Additional comments about lab report:** Fourteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-221213-R1 | 32203536001 | PFBA |
| SP1-02-T1-221213-R1 | 32203536001 | PFOA |
| SP1-02-T1-221213-R1 | 32203536001 | PFOS |
| SP2-01-EFF1-221213-R1 | 32203536002 | PFBA |
| SP2-01-EFF1-221213-R1 | 32203536002 | PFOA |
| SP2-01-EFF1-221213-R1 | 32203536002 | PFOS |
| SP2-01-EFF1-221213-R2 | 32203536003 | PFBA |
| SP2-01-EFF1-221213-R2 | 32203536003 | PFOA |
| SP2-01-EFF1-221213-R2 | 32203536003 | PFOS |
| SP4-01-T4-221213-R1 (15mL) | 32203536004 | PFOA |
| SP4-01-T4-221213-R1 (15mL) | 32203536004 | PFOS |
| SP2-01-EFF1-221213-R3 | 32203536005 | PFBA |
| SP2-01-EFF1-221213-R3 | 32203536005 | PFOA |
| SP2-01-EFF1-221213-R3 | 32203536005 | PFOS |

**10. Additional comments about lab report:** Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32203536 Report date (mm/dd/yyyy): 12/19/2022

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | LCSD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-221213-R1 (15mL) has elevated RLs due to the sample arriving with a lower sample size. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32203699**

**10. Additional comments about lab report:** Fifteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-01-EFF2-221228-R4 | 32203699001 | PFBA |
| SP2-01-EFF2-221228-R4 | 32203699001 | PFOA |
| SP2-01-EFF2-221228-R4 | 32203699001 | PFOS |
| SP2-01-EFF1-221228-R5 | 32203699002 | PFBA |
| SP2-01-EFF1-221228-R5 | 32203699002 | PFOA |
| SP2-01-EFF1-221228-R5 | 32203699002 | PFOS |
| SP2-01-EFF1-221229-R6 | 32203699003 | PFBA |
| SP2-01-EFF1-221229-R6 | 32203699003 | PFOA |
| SP2-01-EFF1-221229-R6 | 32203699003 | PFOS |
| SP2-01-EFF1-221229-R7 | 32203699004 | PFBA |
| SP2-01-EFF1-221229-R7 | 32203699004 | PFOA |
| SP2-01-EFF1-221229-R7 | 32203699004 | PFOS |
| SP2-01-EFF1-221229-R8 | 32203699005 | PFBA |
| SP2-01-EFF1-221229-R8 | 32203699005 | PFOA |
| SP2-01-EFF1-221229-R8 | 32203699005 | PFOS |

**10. Additional comments about lab report:** Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32203699 Report date (mm/dd/yyyy): 1/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300138**

**Labeled Analogs - surrogates (Section 4Bii):** Labeled analog exceedances and qualifications are summarized in the table below. Results for the associated non-labeled analytes were not reported. No qualifiers.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| T4-DRUM-011223 | 32300138009 | 13C2-4:2 FTS | 156 | No Qualifier |
| T4-DRUM-011223 | 32300138009 | 13C2-6:2 FTS | 159 | No Qualifier |
| T4-DRUM-011223 | 32300138009 | 13C2-8:2 FTS | 155 | No Qualifier |

**10. Additional comments about lab report:** Twenty-five results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230111-R9 | 32300138001 | PFBA |
| SP1-02-T1-230111-R9 | 32300138001 | PFOA |
| SP1-02-T1-230111-R9 | 32300138001 | PFOS |
| SP2-01-EFF1-230111-R9 | 32300138002 | PFBA |
| SP2-01-EFF1-230111-R9 | 32300138002 | PFOA |
| SP2-01-EFF1-230111-R9 | 32300138002 | PFOS |
| SP2-01-EFF1-230111-R10 | 32300138003 | PFBA |
| SP2-01-EFF1-230111-R10 | 32300138003 | PFOA |
| SP2-01-EFF1-230111-R10 | 32300138003 | PFOS |
| SP2-01-EFF1-230112-R11 | 32300138004 | PFBA |
| SP2-01-EFF1-230112-R11 | 32300138004 | PFOA |
| SP2-01-EFF1-230112-R11 | 32300138004 | PFOS |
| SP3-01-T3-230111-R9 | 32300138005 | PFOA |
| SP3-01-T3-230111-R9 | 32300138005 | PFOS |
| SP2-01-EFF1-230113-R12 | 32300138006 | PFBA |
| SP2-01-EFF1-230113-R12 | 32300138006 | PFOA |
| SP2-01-EFF1-230113-R12 | 32300138006 | PFOS |
| SP1-02-T1-230112-R11 | 32300138007 | PFBA |
| SP1-02-T1-230112-R11 | 32300138007 | PFOA |
| SP1-02-T1-230112-R11 | 32300138007 | PFOS |
| SP2-01-EFF2-230112-R11 | 32300138008 | PFBA |
| SP2-01-EFF2-230112-R11 | 32300138008 | PFOA |
| SP2-01-EFF2-230112-R11 | 32300138008 | PFOS |
| T4-DRUM-011223 | 32300138009 | PFOA |
| T4-DRUM-011223 | 32300138009 | PFOS |

**10. Additional comments about lab report:** The case narrative indicates that the Internal Standard recoveries for 13C2-PFOA and 13C3-PFBA are greater than +50% relative to the ICAL for sample SP3-01-T3-230111-R9 (32300138005) which may cause a high bias for associated compounds in the sample. Associated results (PFOA, PFHpA, and PFBA) are qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300138 Report date (mm/dd/yyyy): 1/23/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/18/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications required. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples SP3-01-T3-230111-R9 (32300138005) and T4-DRUM-011223 (32300138009) have elevated RLs due to sample arriving with lower sample size. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/18/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300228**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| T4-DRUM-011823 | 32300228006 | 13C2-4:2 FTS | 238 | Associated analytes not reported |
| T4-DRUM-011823 | 32300228006 | 13C2-6:2 FTS | 245 | |
| T4-DRUM-011823 | 32300228006 | 13C2-8:2 FTS | 236 | |
| T4-DRUM-011823 | 32300228006 | 13C3-PFBS | 204 | J- |
| T4-DRUM-011823 | 32300228006 | 13C3-PFHxS | 207 | J- |

**10. Additional comments about lab report:** Twenty-two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230116-R13 | 32300228001 | PFBA |
| SP1-02-T1-230116-R13 | 32300228001 | PFOA |
| SP1-02-T1-230116-R13 | 32300228001 | PFOS |
| SP2-01-EFF1-230116-R13 | 32300228003 | PFBA |
| SP2-01-EFF1-230116-R13 | 32300228003 | PFOA |
| SP2-01-EFF1-230116-R13 | 32300228003 | PFOS |
| SP2-01-EFF1-230117-R14 | 32300228004 | PFBA |
| SP2-01-EFF1-230117-R14 | 32300228004 | PFOA |
| SP2-01-EFF1-230117-R14 | 32300228004 | PFOS |
| SP2-01-EFF1-230118-R15 | 32300228005 | PFBA |
| SP2-01-EFF1-230118-R15 | 32300228005 | PFOA |
| SP2-01-EFF1-230118-R15 | 32300228005 | PFOS |
| SP2-01-EFF1-230120-R16 | 32300228007 | PFBA |
| SP2-01-EFF1-230120-R16 | 32300228007 | PFOA |
| SP2-01-EFF1-230120-R16 | 32300228007 | PFOS |
| SP2-01-EFF2-230116-R13 | 32300228002 | PFBA |
| SP2-01-EFF2-230116-R13 | 32300228002 | PFOA |
| SP2-01-EFF2-230116-R13 | 32300228002 | PFOS |
| T4-DRUM-011823 | 32300228006 | PFDA |
| T4-DRUM-011823 | 32300228006 | PFHxS |
| T4-DRUM-011823 | 32300228006 | PFOA |
| T4-DRUM-011823 | 32300228006 | PFOS |

**10. Additional comments about lab report:** Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300228 Report date (mm/dd/yyyy): 1/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample T4-DRUM-011823 (32300228006-A) has elevated RLs due to sample arriving with lower sample size. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300312**

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230126-R17 | 32300312001 | PFBA |
| SP1-02-T1-230126-R17 | 32300312001 | PFOA |
| SP1-02-T1-230126-R17 | 32300312001 | PFOS |
| SP2-02-EFF1-230126-R17 | 32300312002 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300312 Report date (mm/dd/yyyy): 1/31/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 4/25/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 4/25/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300360**

**Labeled Analogs - surrogates (Section 4Bii):** Labeled analog exceedances and qualifications are summarized in the table below. Results for the associated non-labeled analytes were not reported. No qualifiers.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP2-02-EFF1-230201-R26 | 32300360009 | 13C2-6:2 FTS | 154 | No Qualifier |
| SP2-02-EFF1-230201-R26 | 32300360009 | 13C2-8:2 FTS | 151 | No Qualifier |
| SP2-02-EFF1-230201-R28 | 32300360011 | 13C2-6:2 FTS | 155 | No Qualifier |

**10. Additional comments about lab report:** Twenty-nine results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230130-R18 | 32300360001 | PFBA |
| SP2-02-EFF1-230130-R19 | 32300360002 | PFBA |
| SP2-02-EFF1-230130-R20 | 32300360003 | PFBA |
| SP2-02-EFF1-230131-R21 | 32300360004 | PFBA |
| SP2-02-EFF1-230131-R21 | 32300360004 | PFOA |
| SP2-02-EFF1-230131-R21 | 32300360004 | PFOS |
| SP2-02-EFF1-230131-R22 | 32300360005 | PFBA |
| SP2-02-EFF1-230131-R22 | 32300360005 | PFOA |
| SP2-02-EFF1-230131-R22 | 32300360005 | PFOS |
| SP2-02-EFF1-230131-R23 | 32300360006 | PFBA |
| SP2-02-EFF1-230131-R23 | 32300360006 | PFOA |
| SP2-02-EFF1-230131-R23 | 32300360006 | PFOS |
| SP2-02-EFF1-230201-R24 | 32300360007 | PFBA |
| SP2-02-EFF1-230201-R24 | 32300360007 | PFOA |
| SP2-02-EFF1-230201-R24 | 32300360007 | PFOS |
| SP2-02-EFF1-230201-R25 | 32300360008 | PFBA |
| SP2-02-EFF1-230201-R25 | 32300360008 | PFOA |
| SP2-02-EFF1-230201-R25 | 32300360008 | PFOS |
| SP2-02-EFF1-230201-R26 | 32300360009 | PFBA |
| SP2-02-EFF1-230201-R26 | 32300360009 | PFOA |
| SP2-02-EFF1-230201-R26 | 32300360009 | PFOS |
| SP2-02-EFF1-230201-R27 | 32300360010 | PFBA |
| SP2-02-EFF1-230201-R27 | 32300360010 | PFOA |
| SP2-02-EFF1-230201-R27 | 32300360010 | PFOS |
| SP2-02-EFF1-230201-R28 | 32300360011 | PFBA |
| SP2-02-EFF1-230202-R29 | 32300360012 | PFBA |
| SP2-02-EFF1-230202-R30 | 32300360013 | PFBA |
| SP2-02-EFF1-230202-R30 | 32300360013 | PFOA |
| SP2-02-EFF1-230202-R30 | 32300360013 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300360 Report date (mm/dd/yyyy): 2/8/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/1/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for exceedances. No qualifications required. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Case narrative comments indicate JS responses for two samples were >150% compared to ICAL, but were within CCV limits. This does not impact sample data as analytes reported and surrogates were within criteria. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/1/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300572**

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230216-R43 | 32300572001 | PFBA |
| SP2-02-EFF1-230217-R44 | 32300572002 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300572 Report date (mm/dd/yyyy): 2/22/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/1/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/1/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300616**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below. Associated sample detects within 10 times the blank concentrations were qualified BJ.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 266630 | PFOS | 0.936 J | Associated sample concentrations within 10 times the blank concentration were qualified BJ:<br><br>SP2-02-EFF1-230221-R49 (32300616008) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | 13C2-6:2 FTS | 347 | Associated analytes not reported |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | 13C2-8:2 FTS | 196 | |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | 13C3-PFHxS | 306 | J- |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | 13C6-PFDA | 24 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266631 | PFOS | 137 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |

**10. Additional comments about lab report:** Fifteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230220-R45 | 32300616001 | PFBA |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFHpA |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFNA |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFDA |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFOA |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFHxS |
| SP4-01-T4-230220-R45 (Vial) | 32300616002 | PFOS |
| SP2-02-EFF1-230220-R46 | 32300616003 | PFBA |
| SP1-02-T1SEC-230220-R46 | 32300616004 | PFBA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1SEC-230220-R46 | 32300616004 | PFOA |
| SP1-02-T1SEC-230220-R46 | 32300616004 | PFOS |
| SP2-02-EFF2-230220-R46 | 32300616005 | PFBA |
| SP2-02-EFF1-230221-R47 | 32300616006 | PFBA |
| SP2-02-EFF1-230221-R48 | 32300616007 | PFBA |
| SP2-02-EFF1-230221-R49 | 32300616008 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300616 Report date (mm/dd/yyyy): 2/27/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | Detects were less than the reporting limit. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for method blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-230220-R45 (Vial) has elevated RLs due to sample arriving with lower sample size; however, six analytes exceeded the instrument calibration for this sample due to high concentrations. So it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300704**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below. Sample concentrations were greater than 10 times the blank concentration and results were acceptable without qualification.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 266903 | PFOA | 2.02 | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch 1-TO-230228 | 32300704001 | 13C2-4:2 FTS | 203 | Not Reported |
| Batch 1-TO-230228 | 32300704001 | 13C2-6:2 FTS | 207 | |
| Batch 1-TO-230228 | 32300704001 | 13C2-8:2 FTS | 205 | |
| Batch 1-TO-230228 | 32300704001 | 13C3-PFBS | 214 | J- |
| Batch 1-TO-230228 | 32300704001 | 13C3-PFHxS | 208 | J- |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-4:2 FTS | 192 | Not Reported |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-6:2 FTS | 199 | |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-8:2 FTS | 200 | |
| Batch 1-TO(D)-230228 | 32300704002 | 13C3-PFBS | 210 | J- |
| Batch 1-TO(D)-230228 | 32300704002 | 13C3-PFHxS | 203 | J- |
| Batch 1-T2-230228 | 32300704003 | 13C2-4:2 FTS | 196 | Not Reported |
| Batch 1-T2-230228 | 32300704003 | 13C2-6:2 FTS | 201 | |
| Batch 1-T2-230228 | 32300704003 | 13C2-8:2 FTS | 201 | |
| Batch 1-T2-230228 | 32300704003 | 13C3-PFBS | 210 | J- |
| Batch 1-T2-230228 | 32300704003 | 13C3-PFHxS | 207 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266904 | PFBA | 146 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFHxA | 132 | 70-130 | |
| | PFHpA | 139 | 70-130 | |
| | PFOA | 355 | 70-130 | |
| | PFBS | 134 | 70-130 | |
| | PFHxS | 131 | 70-130 | |
| | PFOS | 139 | 70-130 | |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266904 | PFuNA | 132 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | PFDoA | 132 | 70-130 | |

**10. Additional comments about lab report:** Twelve results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch 1-TO-230228 | 32300704001 | PFOA |
| Batch 1-TO-230228 | 32300704001 | PFHxS |
| Batch 1-TO-230228 | 32300704001 | PFOS |
| Batch 1-TO(D)-230228 | 32300704002 | PFOA |
| Batch 1-TO(D)-230228 | 32300704002 | PFHxS |
| Batch 1-TO(D)-230228 | 32300704002 | PFOS |
| Batch 1-T2-230228 | 32300704003 | PFOA |
| Batch 1-T2-230228 | 32300704003 | PFHxS |
| Batch 1-T2-230228 | 32300704003 | PFOS |
| Batch 1-T8-230301 | 32300704004 | PFOA |
| Batch 1-T8-230301 | 32300704004 | PFHxS |
| Batch 1-T8-230301 | 32300704004 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300704 Report date (mm/dd/yyyy): 3/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicate:<br>Batch 1-TO-230228 / Batch 1-TO(D)-230228 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples had elevated RLs due to sample arriving with lower sample size. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300705**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below. Sample concentrations were greater than 10 times the blank concentration and results were acceptable without qualification.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 266903 | PFOA | 2.02 | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with severely low recoveries were rejected (R). Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230301-R53 | 32300705006 | 13C2-4:2 FTS | 0 | Not Reported |
| SP4-01-T4-230301-R53 | 32300705006 | 13C2-6:2 FTS | 0 | |
| SP4-01-T4-230301-R53 | 32300705006 | 13C3-PFBS | 0 | R |
| SP4-01-T4-230301-R53 | 32300705006 | 13C3-PFHxS | 233 | J- |
| SP4-01-T4-230301-R53 | 32300705006 | 13C6-PFDA | 9 | R |
| SP4-01-T4-230301-R53 | 32300705006 | 13C7-PFuNA | 0 | R |
| SP4-01-T4-230301-R53 | 32300705006 | 13C8-PFOA | 24.9 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266904 | PFBA | 146 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFHxA | 132 | 70-130 | |
| | PFHpA | 139 | 70-130 | |
| | PFOA | 355 | 70-130 | |
| | PFuNA | 132 | 70-130 | |
| | PFBS | 134 | 70-130 | |
| | PFHxS | 131 | 70-130 | |
| | PFOS | 139 | 70-130 | |
| | PFDoA | 132 | 70-130 | Associated results were nondetect and were acceptable without qualification. |

**10. Additional comments about lab report:** Fifteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230224-R50 | 32300705001 | PFBA |
| SP2-02-EFF1-230227-R51 | 32300705002 | PFBA |
| SP1-02-T1-230227-R51 | 32300705003 | PFBA |
| SP1-02-T1-230227-R51 | 32300705003 | PFOA |
| SP1-02-T1-230227-R51 | 32300705003 | PFOS |
| SP2-02-EFF1-230228-R52 | 32300705004 | PFBA |
| SP2-02-EFF1-230301-R53 | 32300705005 | PFBA |
| SP4-01-T4-230301-R53 | 32300705006 | PFHpA |
| SP4-01-T4-230301-R53 | 32300705006 | PFOA |
| SP4-01-T4-230301-R53 | 32300705006 | PFNA |
| SP4-01-T4-230301-R53 | 32300705006 | PFDA |
| SP4-01-T4-230301-R53 | 32300705006 | PFHxS |
| SP4-01-T4-230301-R53 | 32300705006 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300705 Report date (mm/dd/yyyy): 3/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-230301-R53 had elevated RLs due to sample arriving with lower sample size. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Three results were rejected (R) due to severely low labeled analog recoveries.**

**Sample 32300705003 was listed on the chain of custody as SP1-02-EFF1-230227-R51 but was reported as SP1-02-T1-230227-R51.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300720**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T24-230303 (50mL) | 32300720001 | 13C2-8:2 FTS | 154 | Not Reported |
| Batch1-T24-230303 (50mL) | 32300720001 | 13C3-PFBS | 162 | J- |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-4:2 FTS | 157 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-6:2 FTS | 168 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-8:2 FTS | 167 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C3-PFBS | 169 | J- |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C3-PFHxS | 154 | J- |

**10. Additional comments about lab report:** Eight results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T24-230303 (50mL) | 32300720001 | PFHpA |
| Batch1-T24-230303 (50mL) | 32300720001 | PFOA |
| Batch1-T24-230303 (50mL) | 32300720001 | PFHxS |
| Batch1-T24-230303 (50mL) | 32300720001 | PFOS |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFHpA |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFOA |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFHxS |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300720 Report date (mm/dd/yyyy): 3/8/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Two samples were listed on the Sample Summary, that were not reported and were not listed on the chain of custody (Batch1-T24-230303 (250mL) and Batch1-T24(D)-230303 |

| | | | | | |
|---|---|---|---|---|---|
| | | | | | (250mL)) |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicates:<br>Batch1-T24-230303 (50mL) / Batch1-T24(D)-230303 (50mL) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch1-T24-230303 (50mL) and Batch1-T24(D)-230303 (50mL) had elevated RLs due to sample arriving with lower sample size. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300721**

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230302-R54 | 32300721001 | PFBA |
| SP2-02-EFF1-230303-R55 | 32300721002 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300721 Report date (mm/dd/yyyy): 3/8/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300755**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below.

| Batch | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 267060 | PFOA | 2.38 | PFOA results were nondetect or greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). The laboratory indicated that the internal standard 13C4-PFOS was not detect due to severe matrix issues for samples Batch1-T28-Scrubber-Condensate and Batch1-T28-GAC-Condensate. Nineteen results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-PFDoA | 224 | J- |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-HFPO-DA | 34.9 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-PFBS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-PFHxS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C4-PFHpA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C5-PFHxA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C6-PFDA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C7-PFuNA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C8-PFOA | 3.7 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C8-PFOS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C9-PFNA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-4:2 FTS | 0 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-PFDoA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-HFPO-DA | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-PFBS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-PFHxS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C4-PFHpA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C5-PFHxA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C6-PFDA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C7-PFuNA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C8-PFOA | 3.3 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C8-PFOS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C9-PFNA | 0 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| Batch 267061 | PFOA | 232 | 70-130 | Associated detections were qualified as estimated biased high (J+), unless previously rejected (R) due to labeled analog recoveries.<br><br>Batch1-T28-Scrubber-Condensate<br>Batch1-T28-GAC-Condensate |
| | PFOS | 334 | 70-130 | |

**10. Additional comments about lab report:** Seventeen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to labeled analog recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T32-230307 | 32300755001 | PFHpA |
| Batch1-T32-230307 | 32300755001 | PFOA |
| Batch1-T32-230307 | 32300755001 | PFHxS |
| Batch1-T32-230307 | 32300755001 | PFOS |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFBA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHxA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHpA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFOA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFDA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHxS |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFOS |
| Batch1-T28-GAC-Condensate | 32300755003 | PFBA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHxA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T28-GAC-Condensate | 32300755003 | PFDA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHxS |
| Batch1-T28-GAC-Condensate | 32300755003 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300755 Report date (mm/dd/yyyy): 3/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections and discussion. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample Batch1-T32-230307 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Nineteen results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300757**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below.

| Batch | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 267060 | PFOA | 2.38 | PFOA results were greater than 10 times the blank concentration. No qualifiers. |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| Batch 267061 | PFOA | 232 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>SP2-02-EFF1-230306-R56<br>SP2-02-EFF1-230307-R56 |
| | PFOS | 334 | 70-130 | |

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230306-R56 | 32300757001 | PFBA |
| SP2-02-EFF1-230307-R56 | 32300757002 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300757 Report date (mm/dd/yyyy): 3/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| | Questions | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | ☒ | ☐ | ☐ | |
| | i. Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections and discussion. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300882**

**10. Additional comments about lab report:** Six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T64-230310 | 32300882001 | PFHpA |
| Batch1-T64-230310 | 32300882001 | PFOA |
| Batch1-T64-230310 | 32300882001 | PFOS |
| Batch1-T64-230310 | 32300882001 | PFHxS |
| Batch1-T100-230320 | 32300882002 | PFOA |
| Batch1-T100-230320 | 32300882002 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300882 Report date (mm/dd/yyyy): 3/24/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch1-T64-230310 and Batch1-T100-230320 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300883**

**10. Additional comments about lab report:** Six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230313-R57 | 32300883001 | PFBA |
| SP1-02-T1-230313-R57 | 32300883001 | PFOA |
| SP1-02-T1-230313-R57 | 32300883001 | PFOS |
| SP2-02-EFF1-230313-R57 | 32300883002 | PFBA |
| SP2-02-EFF1-230315-R57 | 32300883003 | PFBA |
| SP2-02-EFF1-230317-R57 | 32300883004 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300883 Report date (mm/dd/yyyy): 3/28/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300960**

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T125-230324 | 32300960001 | PFOA |
| Batch1-T125-230324 | 32300960001 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300960 Report date (mm/dd/yyyy): 3/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample Batch1-T125-230324 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301274**

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 268310 | PFBA | 137 | 70-130 | Associated detections were qualified as estimated biased high (J+).<br><br>SP2-02-EFF1-230419-R4<br>SP2-02-EFF1-230420-R3-2<br>SP1-02-T1-230420-R3-2 |
| | PFPeA | 140 | 70-130 | |
| | PFHpA | 138 | 70-130 | |
| | PFHxS | 133 | 70-130 | |

**10. Additional comments about lab report:** Seven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230419-R4 | 32301274001 | PFBA |
| SP2-02-EFF1-230420-R3-2 | 32301274002 | PFBA |
| SP2-02-EFF1-230420-R3-2 | 32301274002 | PFOA |
| SP2-02-EFF1-230420-R3-2 | 32301274002 | PFOS |
| SP1-02-T1-230420-R3-2 | 32301274003 | PFBA |
| SP1-02-T1-230420-R3-2 | 32301274003 | PFOA |
| SP1-02-T1-230420-R3-2 | 32301274003 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301274 Report date (mm/dd/yyyy): 4/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301394**

**10. Additional comments about lab report:** Eighteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230425-R7 | 32301394001 | PFBA |
| SP1-02-T1-230425-R7 | 32301394001 | PFOA |
| SP1-02-T1-230425-R7 | 32301394001 | PFOS |
| SP2-02-EFF1-230425-R7a | 32301394002 | PFBA |
| SP2-02-EFF1-230425-R7a | 32301394002 | PFOA |
| SP2-02-EFF1-230425-R7a | 32301394002 | PFOS |
| SP2-02-EFF1-230425-R7b | 32301394003 | PFBA |
| SP2-02-EFF1-230425-R7b | 32301394003 | PFOA |
| SP2-02-EFF1-230425-R7b | 32301394003 | PFOS |
| SP1-02-T1-230421-R4-2 | 32301394004 | PFBA |
| SP1-02-T1-230421-R4-2 | 32301394004 | PFOA |
| SP1-02-T1-230421-R4-2 | 32301394004 | PFOS |
| SP2-02-EFF1-230421-R4a-2 | 32301394005 | PFBA |
| SP2-02-EFF1-230421-R4a-2 | 32301394005 | PFOA |
| SP2-02-EFF1-230421-R4a-2 | 32301394005 | PFOS |
| SP2-02-EFF1-230421-R4b-2 | 32301394006 | PFBA |
| SP2-02-EFF1-230421-R4b-2 | 32301394006 | PFOA |
| SP2-02-EFF1-230421-R4b-2 | 32301394006 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301394 Report date (mm/dd/yyyy): 5/9/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at 8.9 °C and above the 6 °C criteria. Results were not qualified, as the sampled were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301481**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). The laboratory indicated that the internal standard 13C4-PFOS (used to quantitate the 13C8-PFOS label compound) failed low due to severe matrix issues for sample SP4-01-T4-230504-R11. One result was rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230504-R11 | 32301481007 | 13C2-4:2 FTS | 615 | Not Reported |
| SP4-01-T4-230504-R11 | 32301481007 | 13C8-PFOA | 15.2 | J+ |
| SP4-01-T4-230504-R11 | 32301481007 | 13C9-PFNA | 20.7 | J- |
| SP4-01-T4-230504-R11 | 32301481007 | 13C6-PFDA | 4.6 | R |
| SP4-01-T4-230504-R11 | 32301481007 | 13C3-PFBS | 608 | J- |

**10. Additional comments about lab report:** Fifteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230501-R9 | 32301481001 | PFBA |
| SP2-02-EFF1-230501-R9 | 32301481001 | PFOA |
| SP2-02-EFF1-230501-R9 | 32301481001 | PFOS |
| SP1-02-T1-230502-R8 | 32301481002 | PFBA |
| SP1-02-T1-230502-R8 | 32301481002 | PFOA |
| SP1-02-T1-230502-R8 | 32301481002 | PFOS |
| SP2-02-EFF1-230502-R8 | 32301481003 | PFBA |
| SP2-02-EFF1-230502-R8 | 32301481003 | PFOA |
| SP2-02-EFF1-230502-R8 | 32301481003 | PFOS |
| SP4-01-T4-230504-R11 | 32301481007 | PFHpA |
| SP4-01-T4-230504-R11 | 32301481007 | PFOA |
| SP4-01-T4-230504-R11 | 32301481007 | PFNA |
| SP4-01-T4-230504-R11 | 32301481007 | PFDA |
| SP4-01-T4-230504-R11 | 32301481007 | PFHxS |
| SP4-01-T4-230504-R11 | 32301481007 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301481 Report date (mm/dd/yyyy): 5/10/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-230504-R11 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**One result was rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301482**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that surrogate recoveries were low for samples Batch2-T8-230502 and Batch2-T24-230504 due to severe matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch2-T0-230501 | 32301482001 | 13C2-4:2 FTS | 162 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C2-6:2 FTS | 158 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C2-8:2 FTS | 160 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C3-PFBS | 172 | J- |
| Batch2-T0-230501 | 32301482001 | 13C3-PFHxS | 165 | J- |
| Batch2-T2-230501 | 32301482002 | 13C2-4:2 FTS | 161 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C2-6:2 FTS | 156 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C2-8:2 FTS | 154 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C3-PFBS | 176 | J- |
| Batch2-T2-230501 | 32301482002 | 13C3-PFHxS | 160 | J- |
| Batch2-T8-230502 | 32301482003 | 13C3-HFPO-DA | 30.4 | Not Reported |
| Batch2-T8-230502 | 32301482003 | 13C4-PFBA | 24.4 | J+ |
| Batch2-T8-230502 | 32301482003 | 13C5-PFPeA | 31.5 | J+ |
| Batch2-T8-230502 | 32301482003 | 13C5-PFHxA | 48.5 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C3-HFPO-DA | 46.2 | Not Reported |
| Batch2-T24-230504 | 32301482004 | 13C4-PFBA | 49.2 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C5-PFPeA | 47.9 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C8-PFOA | 43.4 | J+ |

**10. Additional comments about lab report:** Thirteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T0-230501 | 32301482001 | PFOA |
| Batch2-T0-230501 | 32301482001 | PFHxS |
| Batch2-T0-230501 | 32301482001 | PFOS |
| Batch2-T2-230501 | 32301482002 | PFOA |
| Batch2-T2-230501 | 32301482002 | PFHxS |
| Batch2-T2-230501 | 32301482002 | PFOS |
| Batch2-T8-230502 | 32301482003 | PFOA |
| Batch2-T8-230502 | 32301482003 | PFHxS |
| Batch2-T8-230502 | 32301482003 | PFOS |
| Batch2-T24-230504 | 32301482004 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T24-230504 | 32301482004 | PFOA |
| Batch2-T24-230504 | 32301482004 | PFHxS |
| Batch2-T24-230504 | 32301482004 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301482 Report date (mm/dd/yyyy): 5/16/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301586**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). One result was rejected (R) due to a severely low labeled compound recovery. The case narrative indicated that the surrogate recovery exceedances for sample SP4-01-T4-230505-R3 were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230505-R3 | 32301586004 | 13C2-4:2 FTS | 483 | Not Reported |
| SP4-01-T4-230505-R3 | 32301586004 | 13C8-PFOA | 18.0 | J+ |
| SP4-01-T4-230505-R3 | 32301586004 | 13C9-PFNA | 36.3 | J+ |
| SP4-01-T4-230505-R3 | 32301586004 | 13C6-PFDA | 5.9 | R |
| SP4-01-T4-230505-R3 | 32301586004 | 13C3-PFBS | 484 | J- |

**10. Additional comments about lab report:** Twelve results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP4-01-T4-230505-R3 | 32301586004 | PFHpA |
| SP4-01-T4-230505-R3 | 32301586004 | PFDA |
| SP4-01-T4-230505-R3 | 32301586004 | PFHxS |
| SP4-01-T4-230505-R3 | 32301586004 | PFOA |
| SP4-01-T4-230505-R3 | 32301586004 | PFNA |
| SP4-01-T4-230505-R3 | 32301586004 | PFOS |
| SP1-02-T1-230507-R3 | 32301586005 | PFBA |
| SP1-02-T1-230507-R3 | 32301586005 | PFOA |
| SP1-02-T1-230507-R3 | 32301586005 | PFOS |
| SP2-02-EFF1-230507-R3 | 32301586006 | PFBA |
| SP3-01-T3-230507-R3 | 32301586007 | PFOA |
| SP3-01-T3-230507-R3 | 32301586007 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301586 Report date (mm/dd/yyyy): 5/16/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples SP3-01-T3-230505-R3, SP4-01-T4-230505-R3, and SP3-01-T3-230507-R3 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for most samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**One result was rejected (R) due to a severely low labeled compound recovery.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301587**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for samples Batch2-T41-230505 and Batch2-T125-GAC-Condensate-230 were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch2-T41-230505 | 32301587002 | 13C8-PFOA | 49.6 | J+ |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-4:2 FTS | 155 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-6:2 FTS | 160 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-8:2 FTS | 158 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C3-PFBS | 160 | J- |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C2-4:2 FTS | 267 | Not Reported |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C2-8:2 FTS | 42.7 | Not Reported |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C8-PFOA | 13.3 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C9-PFNA | 40.8 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C6-PFDA | 14.4 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C3-PFBS | 270 | J- |

**10. Additional comments about lab report:** Thirty-six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T41-230505 | 32301587002 | PFBA |
| Batch2-T41-230505 | 32301587002 | PFHxA |
| Batch2-T41-230505 | 32301587002 | PFHpA |
| Batch2-T41-230505 | 32301587002 | PFOA |
| Batch2-T41-230505 | 32301587002 | PFHxS |
| Batch2-T41-230505 | 32301587002 | PFOS |
| Batch2-T64-230506 | 32301587003 | PFHpA |
| Batch2-T64-230506 | 32301587003 | PFOA |
| Batch2-T64-230506 | 32301587003 | PFHxS |
| Batch2-T64-230506 | 32301587003 | PFOS |
| Batch2-T100-230508 | 32301587004 | PFBA |
| Batch2-T100-230508 | 32301587004 | PFHxA |
| Batch2-T100-230508 | 32301587004 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T100-230508 | 32301587004 | PFOA |
| Batch2-T100-230508 | 32301587004 | PFHxS |
| Batch2-T100-230508 | 32301587004 | PFOS |
| Batch2-T125-230510 | 32301587005 | PFBA |
| Batch2-T125-230510 | 32301587005 | PFHxA |
| Batch2-T125-230510 | 32301587005 | PFHpA |
| Batch2-T125-230510 | 32301587005 | PFOA |
| Batch2-T125-230510 | 32301587005 | PFHxS |
| Batch2-T125-230510 | 32301587005 | PFOS |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFOA |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFHxS |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFOS |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFHpA |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFOA |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFHxS |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFOS |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFBA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFHpA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFOA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFNA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFDA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFHxS |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301587 Report date (mm/dd/yyyy): 5/23/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch2-T0-Scrubber-230501, Batch2-T64-230506, Batch2-T125-Scrubber-230510, Batch2-T125-Scrubber-Condensat, and Batch2-T125-GAC-Condensate-230 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for most samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301771**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature greater than the 6 °C criteria at 23.1°C. Associated detects associated were qualified as estimated biased low (J-), and the reporting limit for nondetects were qualified as estimated (UJ).

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the continuing calibration verification (CCV) bracketing the samples (6/2/2023 12:56) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Three results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances for 7 of the 8 samples were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to probable matrix interference for most samples. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-TO-230518 | 32301771001 | 13C2-4:2 FTS | 303 | Not Reported |
| Batch3-TO-230518 | 32301771001 | 13C2-6:2 FTS | 169 | Not Reported |
| Batch3-TO-230518 | 32301771001 | 13C8-PFOA | 28.3 | J+ |
| Batch3-TO-230518 | 32301771001 | 13C6-PFDA | 12.9 | J+ |
| Batch3-TO-230518 | 32301771001 | 13C3-PFBS | 311 | J- |
| Batch3-TO-230518 | 32301771001 | 13C3-PFHxS | 192 | J- |
| Batch3-TO(D)-230518 | 32301771002 | 13C2-4:2 FTS | 280 | Not Reported |
| Batch3-TO(D)-230518 | 32301771002 | 13C2-6:2 FTS | 161 | Not Reported |
| Batch3-TO(D)-230518 | 32301771002 | 13C8-PFOA | 29.4 | J+ |
| Batch3-TO(D)-230518 | 32301771002 | 13C6-PFDA | 13.8 | J+ |
| Batch3-TO(D)-230518 | 32301771002 | 13C3-PFBS | 291 | J- |
| Batch3-TO(D)-230518 | 32301771002 | 13C3-PFHxS | 187 | J- |
| Batch3-T2-230518 | 32301771003 | 13C2-4:2 FTS | 156 | Not Reported |
| Batch3-T2-230518 | 32301771003 | 13C2-8:2 FTS | 159 | Not Reported |
| Batch3-T2-230518 | 32301771003 | 13C2-PFDoA | 160 | J- |
| Batch3-T2-230518 | 32301771003 | 13C3-PFBS | 157 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T8-230518 | 32301771004 | 13C2-4:2 FTS | 196 | Not Reported |
| Batch3-T8-230518 | 32301771004 | 13C2-8:2 FTS | 178 | Not Reported |
| Batch3-T8-230518 | 32301771004 | 13C8-PFOA | 31.2 | J+ |
| Batch3-T8-230518 | 32301771004 | 13C6-PFDA | 35.1 | J+ |
| Batch3-T8-230518 | 32301771004 | 13C3-PFBS | 194 | J- |
| Batch3-T24-230522 | 32301771005 | 13C2-4:2 FTS | 229 | Not Reported |
| Batch3-T24-230522 | 32301771005 | 13C4-PFHpA | 32.1 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C8-PFOA | 11.0 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C9-PFNA | 25.9 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C6-PFDA | 6.2 | R |
| Batch3-T24-230522 | 32301771005 | 13C3-PFBS | 260 | J- |
| Batch3-T24(D)-230522 | 32301771006 | 13C2-4:2 FTS | 294 | Not Reported |
| Batch3-T24(D)-230522 | 32301771006 | 13C4-PFHpA | 34.3 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C8-PFOA | 12.2 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C9-PFNA | 27;0 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C6-PFDA | 6.1 | R |
| Batch3-T24(D)-230522 | 32301771006 | 13C3-PFBS | 338 | J- |
| Batch3-T32-230522 | 32301771007 | 13C2-4:2 FTS | 186 | Not Reported |
| Batch3-T32-230522 | 32301771007 | 13C2-8:2 FTS | 37.1 | Not Reported |
| Batch3-T32-230522 | 32301771007 | 13C4-PFHpA | 31.2 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C8-PFOA | 10.6 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C9-PFNA | 22.5 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C6-PFDA | 5.4 | R |
| Batch3-T32-230522 | 32301771007 | 13C3-PFBS | 212 | J- |
| Batch3-T32-230522 | 32301771007 | 13C8-PFOS | 48.8 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C2-8:2 FTS | 163 | Not Reported |
| Batch3-T66-230524 | 32301771008 | 13C4-PFHpA | 48.3 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C8-PFOA | 22.9 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C6-PFDA | 42.1 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C2-PFDoA | 155 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOA | 222 | 70-130 | |
| | PFOS | 870 | 70-130 | |

**10. Additional comments about lab report:** Fifty-three results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-TO-230518 | 32301771001 | PFHpA |
| Batch3-TO-230518 | 32301771001 | PFOA |
| Batch3-TO-230518 | 32301771001 | PFNA |
| Batch3-TO-230518 | 32301771001 | PFDA |
| Batch3-TO-230518 | 32301771001 | PFHxS |
| Batch3-TO-230518 | 32301771001 | PFOS |
| Batch3-TO(D)-230518 | 32301771002 | PFHpA |
| Batch3-TO(D)-230518 | 32301771002 | PFOA |
| Batch3-TO(D)-230518 | 32301771002 | PFNA |
| Batch3-TO(D)-230518 | 32301771002 | PFDA |
| Batch3-TO(D)-230518 | 32301771002 | PFHxS |
| Batch3-TO(D)-230518 | 32301771002 | PFOS |
| Batch3-T2-230518 | 32301771003 | PFHpA |
| Batch3-T2-230518 | 32301771003 | PFOA |
| Batch3-T2-230518 | 32301771003 | PFHxS |
| Batch3-T2-230518 | 32301771003 | PFOS |
| Batch3-T8-230518 | 32301771004 | PFHpA |
| Batch3-T8-230518 | 32301771004 | PFOA |
| Batch3-T8-230518 | 32301771004 | PFNA |
| Batch3-T8-230518 | 32301771004 | PFHxS |
| Batch3-T8-230518 | 32301771004 | PFOS |
| Batch3-T24-230522 | 32301771005 | PFBA |
| Batch3-T24-230522 | 32301771005 | PFHxA |
| Batch3-T24-230522 | 32301771005 | PFHpA |
| Batch3-T24-230522 | 32301771005 | PFOA |
| Batch3-T24-230522 | 32301771005 | PFNA |
| Batch3-T24-230522 | 32301771005 | PFDA |
| Batch3-T24-230522 | 32301771005 | PFHxS |
| Batch3-T24-230522 | 32301771005 | PFOS |
| Batch3-T24(D)-230522 | 32301771006 | PFBA |
| Batch3-T24(D)-230522 | 32301771006 | PFHxA |
| Batch3-T24(D)-230522 | 32301771006 | PFHpA |
| Batch3-T24(D)-230522 | 32301771006 | PFOA |
| Batch3-T24(D)-230522 | 32301771006 | PFNA |
| Batch3-T24(D)-230522 | 32301771006 | PFDA |
| Batch3-T24(D)-230522 | 32301771006 | PFHxS |
| Batch3-T24(D)-230522 | 32301771006 | PFOS |
| Batch3-T32-230522 | 32301771007 | PFBA |
| Batch3-T32-230522 | 32301771007 | PFHxA |
| Batch3-T32-230522 | 32301771007 | PFHpA |
| Batch3-T32-230522 | 32301771007 | PFOA |
| Batch3-T32-230522 | 32301771007 | PFNA |
| Batch3-T32-230522 | 32301771007 | PFDA |
| Batch3-T32-230522 | 32301771007 | PFHxS |
| Batch3-T32-230522 | 32301771007 | PFOS |
| Batch3-T66-230524 | 32301771008 | PFBA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T66-230524 | 32301771008 | PFHxA |
| Batch3-T66-230524 | 32301771008 | PFHpA |
| Batch3-T66-230524 | 32301771008 | PFOA |
| Batch3-T66-230524 | 32301771008 | PFNA |
| Batch3-T66-230524 | 32301771008 | PFDA |
| Batch3-T66-230524 | 32301771008 | PFHxS |
| Batch3-T66-230524 | 32301771008 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

Results with both high and low bias are qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301771 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 23.1 °C and above the 6 °C criteria. Results for samples received at temperatures > 10 °C were qualfied as smmarized in the attached data qualification summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicates:<br>Batch3-TO-230518 / Batch3-TO(D)-230518<br>Batch3-T24-230522 / Batch3-T24(D)-230522 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Three results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301772**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature greater than the 6 °C criteria at 23.1°C. Associated detects associated were qualified as estimated biased low (J-), and the reporting limit for nondetects were qualified as estimated (UJ).

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the continuing calibration verification (CCV) bracketing the samples (6/2/2023 12:56) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for sample SP4-01-T4-230517 were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to matrix for this sample. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230517 | 32301772004 | 13C2-4:2 FTS | 322 | Not Reported |
| SP4-01-T4-230517 | 32301772004 | 13C2-6:2 FTS | 177 | Not Reported |
| SP4-01-T4-230517 | 32301772004 | 13C8-PFOA | 27.5 | J+ |
| SP4-01-T4-230517 | 32301772004 | 13C6-PFDA | 11.7 | J+ |
| SP4-01-T4-230517 | 32301772004 | 13C3-PFBS | 337 | J- |
| SP4-01-T4-230517 | 32301772004 | 13C3-PFHxS | 205 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOA | 222 | 70-130 | |
| | PFOS | 870 | 70-130 | |

**10. Additional comments about lab report:** Eleven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230516-R13 | 32301772001 | PFBA |
| SP1-02-T1-230516-R13 | 32301772001 | PFOA |
| SP1-02-T1-230516-R13 | 32301772001 | PFOS |
| SP2-02-EFF1-230516-R13a | 32301772002 | PFBA |
| SP2-02-EFF1-230516-R13b | 32301772003 | PFBA |
| SP4-01-T4-230517 | 32301772004 | PFHpA |
| SP4-01-T4-230517 | 32301772004 | PFOA |
| SP4-01-T4-230517 | 32301772004 | PFNA |
| SP4-01-T4-230517 | 32301772004 | PFDA |
| SP4-01-T4-230517 | 32301772004 | PFHxS |
| SP4-01-T4-230517 | 32301772004 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

Results with both high and low bias are qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301772 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 23.1 °C and above the 6 °C criteria. Results for samples received at temperatures > 10 °C were qualfied as smmarized in the attached data qualification summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-230517 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for this sample, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301797**

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the 6/2/2023 12:56 continuing calibration verification (CCV) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Twelve results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to probable matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T98-230525 | 32301797001 | 13C2-6:2 FTS | 23.0 | Not Reported |
| Batch3-T98-230525 | 32301797001 | 13C2-8:2 FTS | 27.8 | Not Reported |
| Batch3-T98-230525 | 32301797001 | 13C5-PFHxA | 15.2 | J+ |
| Batch3-T98-230525 | 32301797001 | 13C4-PFHpA | 6.4 | R |
| Batch3-T98-230525 | 32301797001 | 13C6-PFDA | 5.8 | R |
| Batch3-T98-230525 | 32301797001 | 13C8-PFOA | 4.3 | R |
| Batch3-T98-230525 | 32301797001 | 13C9-PFNA | 5.9 | R |
| Batch3-T98-230525 | 32301797001 | 13C3-PFHxS | 30.6 | J+ |
| Batch3-T120-230526 | 32301797002 | 13C2-6:2 FTS | 29.1 | Not Reported |
| Batch3-T120-230526 | 32301797002 | 13C2-8:2 FTS | 27.2 | Not Reported |
| Batch3-T120-230526 | 32301797002 | 13C5-PFHxA | 16.3 | J+ |
| Batch3-T120-230526 | 32301797002 | 13C4-PFHpA | 5.9 | R |
| Batch3-T120-230526 | 32301797002 | 13C8-PFOA | 4.2 | R |
| Batch3-T120-230526 | 32301797002 | 13C9-PFNA | 5.7 | R |
| Batch3-T120-230526 | 32301797002 | 13C6-PFDA | 6.5 | R |
| Batch3-T120-230526 | 32301797002 | 13C3-PFBS | 251 | J- |
| Batch3-T120-230526 | 32301797002 | 13C3-PFHxS | 40.2 | J+ |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C2-4:2 FTS | 1560 | Not Reported |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C2-8:2 FTS | 21.9 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C5-PFHxA | 42.9 | J+ |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C4-PFHpA | 3.6 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C8-PFOA | 1.6 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C9-PFNA | 1.7 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C6-PFDA | 4.3 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C3-PFBS | 1070 | J- |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C3-PFHxS | 46.9 | J+ |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C2-4:2 FTS | 180 | Not Reported |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C8-PFOA | 31.1 | J+ |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C2-PFDoA | 154 | No Qualifier |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C3-PFBS | 187 | No Qualifier |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C2-4:2 FTS | 249 | Not Reported |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C8-PFOA | 13.6 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C9-PFNA | 43.5 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C6-PFDA | 21.3 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C3-PFBS | 268 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOA | 222 | 70-130 | |
| | PFOS | 870 | 70-130 | |

**10. Additional comments about lab report:** Eleven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T98-230525 | 32301797001 | PFBA |
| Batch3-T98-230525 | 32301797001 | PFPeA |
| Batch3-T98-230525 | 32301797001 | PFHpA |
| Batch3-T98-230525 | 32301797001 | PFHxA |
| Batch3-T98-230525 | 32301797001 | PFOA |
| Batch3-T98-230525 | 32301797001 | PFNA |
| Batch3-T98-230525 | 32301797001 | PFDA |
| Batch3-T98-230525 | 32301797001 | PFHxS |
| Batch3-T98-230525 | 32301797001 | PFOS |
| Batch3-T120-230526 | 32301797002 | PFBA |
| Batch3-T120-230526 | 32301797002 | PFPeA |
| Batch3-T120-230526 | 32301797002 | PFHpA |
| Batch3-T120-230526 | 32301797002 | PFHxA |
| Batch3-T120-230526 | 32301797002 | PFOA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T120-230526 | 32301797002 | PFNA |
| Batch3-T120-230526 | 32301797002 | PFDA |
| Batch3-T120-230526 | 32301797002 | PFOS |
| Batch3-T120-230526 | 32301797002 | PFHxS |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFHpA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFOA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFNA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFDA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFHxS |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFOS |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFHpA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFOA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFNA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFHxS |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFOS |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFHpA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFOA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFDA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFNA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFHxS |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301797 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch3-T120-Scrubber-230526 and Batch3-T120-GAC-Condensate-230 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for this sample, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Twelve results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301798**

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the 6/2/2023 12:56 continuing calibration verification (CCV) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | PFOA | 222 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>MW23C-GW-56-60-01-052523 |
| | PFOS | 870 | 70-130 | |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301798 Report date (mm/dd/yyyy): 6/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301900**

**10. Additional comments about lab report:** Six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| MW23C-GW-56-60-03-052523 | 32301900002 | PFOS |
| MW23A-GW-422-432-01-060223 | 32301900003 | PFBA |
| MW23A-GW-422-432-01-060223 | 32301900003 | PFOS |
| MW23B-GW-299-309-01-060523 | 32301900004 | PFBA |
| MW23B-GW-299-309-01-060523 | 32301900004 | PFOA |
| MW23B-GW-299-309-01-060523 | 32301900004 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

# Laboratory data review checklist

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301900 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 9.6 °C and above the 6 °C criteria. Results were acceptable without qualification as the samples were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301901**

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230606-R1 | 32301901001 | PFBA |
| SP2-01-EFF1-230606-R1 | 32301901002 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301901 Report date (mm/dd/yyyy): 6/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 9.6 °C and above the 6 °C criteria. Results were acceptable without qualification as the samples were received at a temperature < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301997**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269995 (Batch HXX3494) | PFOS | 3.43 | Associated sample concentrations within 10 times the blank concentration were qualified B:<br><br>SP1-02-T1-230609-R2<br>SP1-02-T1-230614-R4<br>SP2-02-EFF1-230609-R2<br>SP2-02-EFF1-230612-R3<br>SP2-02-EFF1-230614-R4 |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for sample T4-RFP-230614 were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to matrix. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| T4-RFP-230614 | 32301997006 | 13C2-4:2 FTS | 376 | Not Reported |
| T4-RFP-230614 | 32301997006 | 13C2-6:2 FTS | 194 | Not Reported |
| T4-RFP-230614 | 32301997006 | 13C2-8:2 FTS | 174 | Not Reported |
| T4-RFP-230614 | 32301997006 | 13C8-PFOA | 26.7 | J+ |
| T4-RFP-230614 | 32301997006 | 13C6-PFDA | 11.9 | J+ |
| T4-RFP-230614 | 32301997006 | 13C3-PFBS | 380 | J- |
| T4-RFP-230614 | 32301997006 | 13C3-PFHxS | 229 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269996 (Batch HXX3494) | PFOS | 135 | 70-130 | Associated detections were qualified as estimated biased high (J+), unless previously qualified B due to laboratory contamination:<br><br>T4-RFP-230614 |

**10. Additional comments about lab report:** Eleven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230609-R2 | 32301997001 | PFBA |
| SP2-02-EFF1-230609-R2 | 32301997002 | PFBA |
| SP2-02-EFF1-230612-R3 | 32301997003 | PFBA |
| SP2-02-EFF1-230614-R4 | 32301997004 | PFBA |
| SP1-02-T1-230614-R4 | 32301997005 | PFBA |
| T4-RFP-230614 | 32301997006 | PFHpA |
| T4-RFP-230614 | 32301997006 | PFOA |
| T4-RFP-230614 | 32301997006 | PFNA |
| T4-RFP-230614 | 32301997006 | PFDA |
| T4-RFP-230614 | 32301997006 | PFHxS |
| T4-RFP-230614 | 32301997006 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301997 Report date (mm/dd/yyyy): 6/21/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for method blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample T4-RFP-230614 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for this sample, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301998**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269995 (Batch HXX3494) | PFOS | 3.43 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| 270009 (Batch HXX3496) | PFOA | 1.66 J | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 8.92 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). One result was rejected (R) due to a severely low labeled compound recovery. The case narrative indicated that the surrogate recovery exceedances for sample Batch4-T0-230612 were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to matrix. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T0-230612 | 32301998001 | 13C2-4:2 FTS | 328 | Not Reported |
| Batch4-T0-230612 | 32301998001 | 13C2-6:2 FTS | 153 | Not Reported |
| Batch4-T0-230612 | 32301998001 | 13C8-PFOA | 22.5 | J+ |
| Batch4-T0-230612 | 32301998001 | 13C6-PFDA | 7.5 | R |
| Batch4-T0-230612 | 32301998001 | 13C3-PFBS | 339 | J- |
| Batch4-T0-230612 | 32301998001 | 13C3-PFHxS | 177 | J- |
| Batch4-T2-230614 | 32301998002 | 13C2-4:2 FTS | 230 | Not Reported |
| Batch4-T2-230614 | 32301998002 | 13C8-PFOA | 22.6 | J+ |
| Batch4-T2-230614 | 32301998002 | 13C6-PFDA | 17.6 | J+ |
| Batch4-T2-230614 | 32301998002 | 13C3-PFBS | 231 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below. The case narrative indicates that the high recoveries are due to probable contamination from high level samples.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269996 (Batch HXX3494) | PFOS | 135 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>Batch4-T0-230612<br>Batch4-T2-230614 |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 270010 (Batch HXX3496) | PFOA | 275 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>Batch4-T1-Scrubber-230614 |
| | PFHxS | 136 | 70-130 | |
| | PFOS | 795 | 70-130 | |

**10. Additional comments about lab report:** Thirteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T0-230612 | 32301998001 | PFHxS |
| Batch4-T0-230612 | 32301998001 | PFHpA |
| Batch4-T0-230612 | 32301998001 | PFOA |
| Batch4-T0-230612 | 32301998001 | PFNA |
| Batch4-T0-230612 | 32301998001 | PFDA |
| Batch4-T0-230612 | 32301998001 | PFOS |
| Batch4-T2-230614 | 32301998002 | PFHpA |
| Batch4-T2-230614 | 32301998002 | PFOA |
| Batch4-T2-230614 | 32301998002 | PFNA |
| Batch4-T2-230614 | 32301998002 | PFDA |
| Batch4-T2-230614 | 32301998002 | PFHxS |
| Batch4-T2-230614 | 32301998002 | PFOS |
| Batch4-T1-Scrubber-230614 | 32301998003 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301998 Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**One result was rejected (R) due to a severely low labeled compound recovery.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302028**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270009 (Batch HXX3496) | PFOA | 1.66 J | Associated sample concentrations were within 10 times the blank concentration and qualified BJ:<br><br>SP2-02-EFF1-230615-R5<br>SP2-02-EFF1-230616-R6 |
| | PFOS | 8.92 | Associated sample concentrations were within 10 times the blank concentration and qualified BJ:<br><br>SP2-02-EFF1-230615-R5 |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances are listed below. The case narrative indicates that the high recoveries are due to probable contamination from high level samples.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 270010 (Batch HXX3496) | PFOA | 275 | 70-130 | Associated sample detects were previously qualified due to laboratory contamination. |
| | PFOS | 795 | 70-130 | |
| | PFHxS | 136 | 70-130 | The associated results were nondetect and did not require qualification. |

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP2-02-EFF1-230615-R5 | 32302028001 | PFBA |
| SP2-02-EFF1-230616-R6 | 32302028002 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302028 Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/7/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 9.5 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for method blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/7/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302031**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270009 (Batch HXX3496) | PFOA | 1.66 J | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 8.92 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Two results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T8-230615 | 32302031001 | 13C2-4:2 FTS | 166 | Not Reported |
| Batch4-T8-230615 | 32302031001 | 13C2-8:2 FTS | 181 | Not Reported |
| Batch4-T8-230615 | 32302031001 | 13C8-PFOA | 33.4 | J+ |
| Batch4-T8-230615 | 32302031001 | 13C3-PFBS | 169 | J- |
| Batch4-T26-230616 | 32302031002 | 13C2-6:2 FTS | 40.8 | Not Reported |
| Batch4-T26-230616 | 32302031002 | 13C3-HFPO-DA | 46.3 | Not Reported |
| Batch4-T26-230616 | 32302031002 | 13C5-PFHxA | 43.8 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C4-PFHpA | 25.8 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C8-PFOA | 7.9 | R |
| Batch4-T26-230616 | 32302031002 | 13C9-PFNA | 22.6 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C6-PFDA | 11.2 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C3-PFHxS | 44.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C2-4:2 FTS | 168 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C2-6:2 FTS | 49.5 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C3-HFPO-DA | 45.9 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C5-PFHxA | 48.5 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C4-PFHpA | 25.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C8-PFOA | 9.0 | R |
| Batch4-T26(D)-230616 | 32302031003 | 13C9-PFNA | 20.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C6-PFDA | 11.4 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C3-PFBS | 198 | J- |
| Batch4-T32-230616 | 32302031004 | 13C4-PFHpA | 37.7 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T32-230616 | 32302031004 | 13C8-PFOA | 14.7 | J+ |
| Batch4-T32-230616 | 32302031004 | 13C6-PFDA | 23.6 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances are listed below. The case narrative indicates that the high recoveries are due to probable contamination from high level samples.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 270010 (Batch HXX3496) | PFOA | 275 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOS | 795 | 70-130 | |
| | PFHxS | 136 | 70-130 | |

**10. Additional comments about lab report:** Thenty-six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to severely low labeled compound recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T8-230615 | 32302031001 | PFHpA |
| Batch4-T8-230615 | 32302031001 | PFHxS |
| Batch4-T8-230615 | 32302031001 | PFOA |
| Batch4-T8-230615 | 32302031001 | PFOS |
| Batch4-T26-230616 | 32302031002 | PFBA |
| Batch4-T26-230616 | 32302031002 | PFHxA |
| Batch4-T26-230616 | 32302031002 | PFHpA |
| Batch4-T26-230616 | 32302031002 | PFOA |
| Batch4-T26-230616 | 32302031002 | PFNA |
| Batch4-T26-230616 | 32302031002 | PFHxS |
| Batch4-T26-230616 | 32302031002 | PFOS |
| Batch4-T26(D)-230616 | 32302031003 | PFBA |
| Batch4-T26(D)-230616 | 32302031003 | PFHxA |
| Batch4-T26(D)-230616 | 32302031003 | PFHpA |
| Batch4-T26(D)-230616 | 32302031003 | PFOA |
| Batch4-T26(D)-230616 | 32302031003 | PFNA |
| Batch4-T26(D)-230616 | 32302031003 | PFHxS |
| Batch4-T26(D)-230616 | 32302031003 | PFOS |
| Batch4-T32-230616 | 32302031004 | PFBA |
| Batch4-T32-230616 | 32302031004 | PFHxA |
| Batch4-T32-230616 | 32302031004 | PFHpA |
| Batch4-T32-230616 | 32302031004 | PFOA |
| Batch4-T32-230616 | 32302031004 | PFNA |
| Batch4-T32-230616 | 32302031004 | PFDA |
| Batch4-T32-230616 | 32302031004 | PFHxS |
| Batch4-T32-230616 | 32302031004 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302031 Report date (mm/dd/yyyy): 6/27/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/7/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 9.5 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Batch4-T26-230616 / Batch4-T26(D)-230616 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Two results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/7/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302108**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270256 (Batch HXX/3507) | PFOA | 2.34 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 2.66 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Thirty-three results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that most surrogate recovery exceedances were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C2-4:2 FTS | 153 | Not Reported |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C2-8:2 FTS | 166 | Not Reported |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C8-PFOA | 49.3 | J+ |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C3-PFBS | 160 | J- |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C3-PFHxS | 153 | J- |
| Batch4-T64-230620 | 32302108002 | 13C2-4:2 FTS | 20.1 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C2-6:2 FTS | 13.5 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C2-8:2 FTS | 18.7 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C3-HFPO-DA | 5.7 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C4-PFBA | 12.4 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C5-PFPeA | 25.6 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C5-PFHxA | 6.6 | R |
| Batch4-T64-230620 | 32302108002 | 13C4-PFHpA | 4.4 | R |
| Batch4-T64-230620 | 32302108002 | 13C8-PFOA | 1.9 | R |
| Batch4-T64-230620 | 32302108002 | 13C9-PFNA | 3.0 | R |
| Batch4-T64-230620 | 32302108002 | 13C6-PFDA | 3.0 | R |
| Batch4-T64-230620 | 32302108002 | 13C7-PFuNA | 20.5 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C2-PFDoA | 30.6 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C3-PFBS | 41.1 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C3-PFHxS | 16.4 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C8-PFOS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C2-4:2 FTS | 0 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T102-230622 | 32302108003 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C3-HFPO-DA | 9.4 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C4-PFBA | 31.2 | J+ |
| Batch4-T102-230622 | 32302108003 | 13C4-PFHpA | 3.6 | R |
| Batch4-T102-230622 | 32302108003 | 13C5-PFHxA | 12.6 | J+ |
| Batch4-T102-230622 | 32302108003 | 13C8-PFOA | 2.1 | R |
| Batch4-T102-230622 | 32302108003 | 13C9-PFNA | 1.8 | R |
| Batch4-T102-230622 | 32302108003 | 13C6-PFDA | 5.0 | R |
| Batch4-T102-230622 | 32302108003 | 13C7-PFuNA | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C2-PFDoA | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C8-PFOS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C3-PFBS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C3-PFHxS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C3-HFPO-DA | 5.4 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C4-PFBA | 16.5 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C5-PFPeA | 49.9 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C5-PFHxA | 10.3 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C4-PFHpA | 4.4 | R |
| Batch4-T125-230623 | 32302108004 | 13C8-PFOA | 1.4 | R |
| Batch4-T125-230623 | 32302108004 | 13C9-PFNA | 1.2 | R |
| Batch4-T125-230623 | 32302108004 | 13C6-PFDA | 2.3 | R |
| Batch4-T125-230623 | 32302108004 | 13C7-PFuNA | 44.8 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C2-PFDoA | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C3-PFBS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C3-PFHxS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C8-PFOS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-HFPO-DA | 263 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C5-PFHxA | 271 | J- |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C4-PFHpA | 46.4 | J+ |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C8-PFOA | 5.2 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C9-PFNA | 2.4 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C6-PFDA | 12.2 | J+ |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C7-PFuNA | 158 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-PFDoA | 249 | J- |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-PFBS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-PFHxS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C8-PFOS | 0 | R |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C2-4:2 FTS | 157 | Not Reported |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C2-8:2 FTS | 182 | Not Reported |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C3-PFBS | 174 | No Qualifier |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C3-PFHxS | 189 | J- |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C2-8:2 FTS | 19.7 | Not Reported |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C4-PFHpA | 20.7 | J+ |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C8-PFOA | 5.3 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C9-PFNA | 3.6 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C6-PFDA | 5.3 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C3-PFBS | 0 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C8-PFOS | 0 | R |

**10. Additional comments about lab report:** Forty-nine results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to severely low labeled compound recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T1-Scrubber-230612 | 32302108001 | PFOA |
| Batch4-T1-Scrubber-230612 | 32302108001 | PFHxS |
| Batch4-T1-Scrubber-230612 | 32302108001 | PFOS |
| Batch4-T64-230620 | 32302108002 | PFBA |
| Batch4-T64-230620 | 32302108002 | PFPeA |
| Batch4-T64-230620 | 32302108002 | PFHpA |
| Batch4-T64-230620 | 32302108002 | PFHxA |
| Batch4-T64-230620 | 32302108002 | PFOA |
| Batch4-T64-230620 | 32302108002 | PFNA |
| Batch4-T64-230620 | 32302108002 | PFDA |
| Batch4-T64-230620 | 32302108002 | PFHxS |
| Batch4-T64-230620 | 32302108002 | PFOS |
| Batch4-T102-230622 | 32302108003 | PFBA |
| Batch4-T102-230622 | 32302108003 | PFPeA |
| Batch4-T102-230622 | 32302108003 | PFHxA |
| Batch4-T102-230622 | 32302108003 | PFHpA |
| Batch4-T102-230622 | 32302108003 | PFOA |
| Batch4-T102-230622 | 32302108003 | PFNA |
| Batch4-T102-230622 | 32302108003 | PFDA |
| Batch4-T102-230622 | 32302108003 | PFHxS |
| Batch4-T102-230622 | 32302108003 | PFOS |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T125-230623 | 32302108004 | PFBA |
| Batch4-T125-230623 | 32302108004 | PFPeA |
| Batch4-T125-230623 | 32302108004 | PFHxA |
| Batch4-T125-230623 | 32302108004 | PFHpA |
| Batch4-T125-230623 | 32302108004 | PFNA |
| Batch4-T125-230623 | 32302108004 | PFOA |
| Batch4-T125-230623 | 32302108004 | PFDA |
| Batch4-T125-230623 | 32302108004 | PFHxS |
| Batch4-T125-230623 | 32302108004 | PFOS |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFHpA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFOA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFNA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFDA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFHxS |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFOS |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFOA |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFDA |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFOS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFBA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFPeA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHxA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHpA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFOA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFNA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFDA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFBS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHxS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302108 Report date (mm/dd/yyyy): 6/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/7/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 8.8 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided.<br>The case narrative indicated that the PFOS CCV recovery was above limits for CCV2, and that low level concentrations may be biased high. PFOS sample concentrations exceeded the instrument calibration range, and were qualified as estimated (J). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Three samples (Batch4-T1-Scrubber-230612, Batch4-T125-Scrubber-230623, and Batch4-T125-GAC-Condensate-230) had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Thirty-three results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302109**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270256 (Batch HXX/3507) | PFOA | 2.34 | Associated sample concentrations that were within 10 times the blank concentration were qualified BJ:<br><br>SP2-02-EFF1-230619-R7<br>SP2-02-EFF1-230623-R8 |
| | PFOS | 2.66 | Associated sample concentrations that were within 10 times the blank concentration were qualified BJ:<br><br>SP2-02-EFF1-230619-R7<br>SP1-02-T1-230623-R8<br>SP2-02-EFF1-230623-R8 |

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230619-R7 | 32302109001 | PFBA |
| SP1-02-T1-230623-R8 | 32302109003 | PFBA |
| SP2-02-EFF1-230619-R7 | 32302109002 | PFBA |
| SP2-02-EFF1-230623-R8 | 32302109004 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302109 Report date (mm/dd/yyyy): 6/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 8.8 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided.<br>The case narrative indicated that the PFOS CCV recovery was above limits for CCV2, and that low level concentrations may be biased high. Low level PFOS concentrations were qualified BJ due to laboratory contamination. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for method blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302359**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the 13C4-PFBA surrogate recovery exceedance and the 13C4-PFOS high internal standard were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP4-01-T4-230712 | 32302359007 | 13C2-4:2 FTS | 193 | Not Reported |
| SP4-01-T4-230712 | 32302359007 | 13C2-6:2 FTS | 179 | Not Reported |
| SP4-01-T4-230712 | 32302359007 | 13C2-8:2 FTS | 189 | Not Reported |
| SP4-01-T4-230712 | 32302359007 | 13C4-PFBA | 17.9 | J+ |
| SP4-01-T4-230712 | 32302359007 | 13C2-PFDoA | 171 | No Qualifier |
| SP4-01-T4-230712 | 32302359007 | 13C7-PFuNA | 155 | No Qualifier |
| SP4-01-T4-230712 | 32302359007 | 13C3-PFBS | 198 | J- |
| SP4-01-T4-230712 | 32302359007 | 13C3-PFHxS | 168 | J- |

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to severely low labeled compound recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230710-R9 | 32302359001 | PFBA |
| SP2-02-EFF1-230710-R9 | 32302359002 | PFBA |
| SP1-02-T1-230711-R10 | 32302359003 | PFBA |
| SP2-02-EFF1-230711-R10 | 32302359004 | PFBA |
| SP1-02-T1-230712-R11 | 32302359005 | PFBA |
| SP2-02-EFF1-230712-R11 | 32302359006 | PFBA |
| SP4-01-T4-230712 | 32302359007 | PFHpA |
| SP4-01-T4-230712 | 32302359007 | PFOA |
| SP4-01-T4-230712 | 32302359007 | PFHxS |
| SP4-01-T4-230712 | 32302359007 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302359 Report date (mm/dd/yyyy): 7/19/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 8.2 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP4-01-T4-230712 had elevated RLs due to a lower sample volume. However, analytes exceeded the instrument calibration for this sample, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 6/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302360**

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302360 Report date (mm/dd/yyyy): 7/20/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 8.2 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302395**

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230713-R12 | 32302395001 | PFBA |
| SP1-02-T1-230717-R13 | 32302395003 | PFBA |
| SP2-02-EFF1-230713-R12 | 32302395002 | PFBA |
| SP2-02-EFF1-230717-R13 | 32302395004 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302395 Report date (mm/dd/yyyy): 7/24/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/12/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/12/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302437**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 271211 (Batch XLC2448) | PFOS | 7.39 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for samples DRUM1-PIKE-RINSE-METHANOL-0720 and DRUM3-PIKE-RINSE-WATER-072023 were due to matrix interference; and that the internal standard 13C4-PFOS was outside control limits due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-4:2 FTS | 263 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-6:2 FTS | 247 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-8:2 FTS | 201 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C6-PFDA | 29.6 | J+ |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C3-PFBS | 283 | No Qualifier |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C3-PFHxS | 272 | J- |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-4:2 FTS | 181 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-6:2 FTS | 167 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-8:2 FTS | 152 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C3-PFBS | 195 | No Qualifier |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C3-PFHxS | 187 | J- |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-HFPO-DA | 45.4 | Not Reported |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C5-PFPeA | 44.2 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C4-PFHpA | 48.9 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C8-PFOA | 45.6 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C9-PFNA | 48.1 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C6-PFDA | 41.4 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-PFBS | 160 | No Qualifier |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-PFHxS | 157 | J- |

**10. Additional comments about lab report:** Eight results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFOA |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFDA |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFHxS |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFOS |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | PFOA |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | PFOS |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | PFOA |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302437 Report date (mm/dd/yyyy): 8/3/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/12/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 7.1 °C, and above the 6 °C criteria. Results were not qualified, as the samples were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | The samples had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/12/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302550**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 271559 (Batch XLC2448) | PFOS | 4.81 | Associated sample concentrations that were within 10 times the blank concentration were qualified BJ:<br><br>SP1-01-INF-230721-R6<br>SP1-02-T1-230721-R6<br>SP1-02-T1SEC-230721-R6<br>SP1-02-T1-230724-R14<br>SP1-02-T1-230725-R15<br>SP1-02-T1-230726-R11B<br>SP2-02-EFF1-230726-R11B |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 271560 | PFOS | 223 | 70-130 | Associated detections were qualified as estimated biased high (J+), unless previously qualified due to laboratory contamination:<br><br>SP3-01-T3-230721-R6 |

**10. Additional comments about lab report:** Eleven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-01-INF-230721-R6 | 32302550001 | PFBA |
| SP1-02-T1-230721-R6 | 32302550002 | PFBA |
| SP2-02-EFF1B-230721-R6 | 32302550003 | PFBA |
| SP1-02-T1SEC-230721-R6 | 32302550004 | PFBA |
| SP2-02-EFF2-230721-R6 | 32302550005 | PFBA |
| SP1-02-T1-230724-R14 | 32302550007 | PFBA |
| SP2-02-EFF1-230724-R146 | 32302550008 | PFBA |
| SP1-02-T1-230725-R15 | 32302550009 | PFBA |
| SP2-02-EFF1B-230725-R15 | 32302550010 | PFBA |
| SP1-02-T1-230726-R11B | 32302550011 | PFBA |
| SP2-02-EFF1-230726-R11B | 32302550012 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302550 Report date (mm/dd/yyyy): 8/3/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/12/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 9.3 °C, and above the 6 °C criteria. Results were not qualified, as the samples were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for method blank detections and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP3-01-T3-230721-R6 had elevated RLs due to a lower sample volume that was used due to suspected high concentrations. High analyte concentrations were reported, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 6/12/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302614**

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230727-R4B | 32302614003 | PFBA |
| SP1-02-T1-230728-R16 | 32302614001 | PFBA |
| SP2-02-EFF1-230727-R4B | 32302614004 | PFBA |
| SP2-02-EFF1B-230728-R16 | 32302614002 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302614 Report date (mm/dd/yyyy): 8/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/12/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/12/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302628**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for sample MW25A-GW-390-400-01-072423 were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW25A-GW-390-400-01-072423 | 32302628001 | 13C3-HFPO-DA | 34.3 | Not Reported |
| MW25A-GW-390-400-01-072423 | 32302628001 | 13C4-PFBA | 39.2 | J+ |
| MW25A-GW-390-400-01-072423 | 32302628001 | 13C5-PFPeA | 32.7 | J+ |
| MW25A-GW-390-400-01-072423 | 32302628001 | 13C5-PFHxA | 41.9 | J+ |
| MW25A-GW-390-400-01-072423 | 32302628001 | 13C4-PFHpA | 47.2 | J+ |

**10. Additional comments about lab report:** One result was quantitated above the instrument calibration range as listed below, and was qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| MW25A-GW-390-400-01-072423 | 32302628001 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302628 Report date (mm/dd/yyyy): 8/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/13/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302760**

**10. Additional comments about lab report:** Ten results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-01-INF-230816-R16A | 32302760016 | PFBA |
| SP1-02-T1-230816-R16A | 32302760017 | PFBA |
| SP2-02-EFF1-230816-R16A | 32302760018 | PFBA |
| SP1-01-INF-230816-R16B | 32302760019 | PFBA |
| SP1-02-T1-230816-R16B | 32302760020 | PFBA |
| SP2-02-EFF1-230816-R16B | 32302760021 | PFBA |
| SP1-02-T1SEC-230816-R16B | 32302760022 | PFBA |
| SP2-02-EFF2-230816-R16B | 32302760023 | PFBA |
| SP3-01-T3-230816 | 32302760024 | PFBA |
| SP3-01-T3-230816 | 32302760024 | PFOA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302760 Report date (mm/dd/yyyy): 8/23/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/13/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | Additional samples were listed on the COCs and sample summary which were not reported in the laboratory report. Only samples 32302760016 through 32302760024 were included, as the other samples were placed on hold.<br>COC page 1 was not signed and dated by the laboratory. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32303168**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+/UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for most samples were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SPI-02-TI-230913-R57 | 32303168001 | 13C3-HFPO-DA | 28.8 | Not Reported |
| SPI-02-TI-230913-R57 | 32303168001 | 13C5-PFPeA | 33.1 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C5-PFHxA | 31.8 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C4-PFHpA | 29.4 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C8-PFOA | 30.0 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C9-PFNA | 33.2 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C6-PFDA | 34.3 | J+ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C7-PFuNA | 36.1 | UJ |
| SPI-02-TI-230913-R57 | 32303168001 | 13C2-PFDoA | 37.5 | UJ |
| SP2-02-EFF1-230913-R57 | 32303168002 | 13C3-HFPO-DA | 31.7 | Not Reported |
| SP2-02-EFF1-230913-R57 | 32303168002 | 13C5-PFPeA | 39.6 | J+ |
| SP2-02-EFF1-230913-R57 | 32303168002 | 13C5-PFHxA | 38.4 | J+ |
| SP2-02-EFF1-230913-R57 | 32303168002 | 13C4-PFHpA | 38.3 | J+ |
| SP2-02-EFF1-230913-R57 | 32303168002 | 13C8-PFOA | 44.0 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C2-PFDoA | 35.8 | UJ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C3-HFPO-DA | 20.5 | Not Reported |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C4-PFBA | 43.7 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C5-PFPeA | 26.6 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C5-PFHxA | 24.9 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C4-PFHpA | 22.0 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C8-PFOA | 23.4 | J+ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C9-PFNA | 26.0 | UJ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C6-PFDA | 29.8 | UJ |
| SP2-02-EFF1-230914-R58 | 32303168004 | 13C7-PFuNA | 32.5 | UJ |
| SPI-02-TI-230915-R59 | 32303168005 | 13C3-HFPO-DA | 43.4 | Not Reported |
| SPI-02-TI-230915-R59 | 32303168005 | 13C5-PFPeA | 48.5 | J+ |
| SPI-02-TI-230915-R59 | 32303168005 | 13C4-PFHpA | 48.7 | J+ |
| SPI-02-TI-230915-R59 | 32303168005 | 13C8-PFOA | 48.1 | J+ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C2-PFDoA | 42.2 | UJ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C3-HFPO-DA | 22.7 | Not Reported |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C4-PFBA | 42.1 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C5-PFPeA | 28.3 | J+ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C5-PFHxA | 27.3 | J+ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C4-PFHpA | 27.4 | J+ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C8-PFOA | 27.9 | J+ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C9-PFNA | 32.1 | UJ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C6-PFDA | 36.4 | UJ |
| SP2-02-EFF1-230915-R59 | 32303168006 | 13C7-PFuNA | 39.4 | UJ |

**10. Additional comments about lab report:** Twelve results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SPI-02-TI-230913-R57 | 32303168001 | PFBA |
| SPI-02-TI-230913-R57 | 32303168001 | PFOA |
| SPI-02-TI-230913-R57 | 32303168001 | PFOS |
| SP2-02-EFF1-230913-R57 | 32303168002 | PFBA |
| SPI-02-TI-230914-R58 | 32303168003 | PFBA |
| SPI-02-TI-230914-R58 | 32303168003 | PFOA |
| SPI-02-TI-230914-R58 | 32303168003 | PFOS |
| SP2-02-EFF1-230914-R58 | 32303168004 | PFBA |
| SPI-02-TI-230915-R59 | 32303168005 | PFBA |
| SPI-02-TI-230915-R59 | 32303168005 | PFOA |
| SPI-02-TI-230915-R59 | 32303168005 | PFOS |
| SP2-02-EFF1-230915-R59 | 32303168006 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32303168 Report date (mm/dd/yyyy): 9/21/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/13/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32303185**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+/UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for most samples were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below. Four PFBA results were rejected (R) due to severely low labeled compound recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP1-02-T1-230918-R60B | 32303185001 | 13C3-HFPO-DA | 41.9 | Not Reported |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C3-HFPO-DA | 13.6 | Not Reported |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C4-PFBA | 43.9 | J+ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C5-PFPeA | 24.8 | J+ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C5-PFHxA | 17.9 | J+ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C4-PFHpA | 14.1 | J+ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C8-PFOA | 16.3 | UJ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C9-PFNA | 21.3 | UJ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C6-PFDA | 28.7 | UJ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C7-PFuNA | 35.2 | UJ |
| SP2-02-EFF1-230918-R60B | 32303185002 | 13C2-PFDoA | 41.6 | UJ |
| SP3-01-T3-230918-R60 | 32303185003 | 13C3-HFPO-DA | 2.1 | Not Reported |
| SP3-01-T3-230918-R60 | 32303185003 | 13C4-PFBA | 4.3 | R |
| SP3-01-T3-230918-R60 | 32303185003 | 13C4-PFHpA | 7.4 | R |
| SP3-01-T3-230918-R60 | 32303185003 | 13C5-PFHxA | 4.6 | R |
| SP3-01-T3-230918-R60 | 32303185003 | 13C5-PFPeA | 2.7 | R |
| SP3-01-T3-230918-R60 | 32303185003 | 13C8-PFOA | 12.3 | J+ |
| SP3-01-T3-230918-R60 | 32303185003 | 13C9-PFNA | 21.3 | UJ |
| SP3-01-T3-230918-R60 | 32303185003 | 13C6-PFDA | 29.9 | J+ |
| SP3-01-T3-230918-R60 | 32303185003 | 13C7-PFuNA | 38.3 | UJ |
| SP3-01-T3-230918-R60 | 32303185003 | 13C2-PFDoA | 44.7 | UJ |
| SP1-02-TISEC-230918-R60B | 32303185004 | 13C3-HFPO-DA | 48.1 | Not Reported |

**10. Additional comments about lab report:** Ten results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230918-R60B | 32303185001 | PFBA |
| SP1-02-T1-230918-R60B | 32303185001 | PFOA |
| SP1-02-T1-230918-R60B | 32303185001 | PFOS |
| SP2-02-EFF1-230918-R60B | 32303185002 | PFBA |
| SP3-01-T3-230918-R60 | 32303185003 | PFOA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP3-01-T3-230918-R60 | 32303185003 | PFOS |
| SP1-02-TISEC-230918-R60B | 32303185004 | PFBA |
| SP1-02-TISEC-230918-R60B | 32303185004 | PFOA |
| SP1-02-TISEC-230918-R60B | 32303185004 | PFOS |
| SP2-02-EFF2-230918-R60B | 32303185005 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32303185 Report date (mm/dd/yyyy): 9/22/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/13/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP3-01-T3-230918-R60 had elevated RLs due to a lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

**Four PFBA results were rejected (R) due to severely low labeled compound recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32303357**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP3-01-T3-230926-R62 | 32303357007 | 13C4-PFBA | 45.7 | J+ |

**10. Additional comments about lab report:** Nineteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-230921-R61A | 32303357001 | PFBA |
| SP1-02-T1-230921-R61A | 32303357001 | PFOA |
| SP1-02-T1-230921-R61A | 32303357001 | PFOS |
| SP2-02-EFF1-230921-R61A | 32303357002 | PFBA |
| SP1-02-T1-230926-R62B | 32303357003 | PFBA |
| SP1-02-T1-230926-R62B | 32303357003 | PFOA |
| SP1-02-T1-230926-R62B | 32303357003 | PFOS |
| SP2-02-EFF1-230926-R62B | 32303357004 | PFBA |
| SP1-02-TISEC-230926-R62B | 32303357005 | PFBA |
| SP1-02-TISEC-230926-R62B | 32303357005 | PFOA |
| SP1-02-TISEC-230926-R62B | 32303357005 | PFOS |
| SP2-02-EFF2-230926-R62B | 32303357006 | PFBA |
| SP3-01-T3-230926-R62 | 32303357007 | PFOA |
| SP3-01-T3-230926-R62 | 32303357007 | PFOS |
| SP2-02-EFF1-230927-R63A | 32303357008 | PFBA |
| SP2-02-EFF1-230928-R64B | 32303357009 | PFBA |
| SP2-02-EFF1-230929-R65A | 32303357010 | PFBA |
| SP1-02-T1-230930-R12 | 32303357011 | PFOS |
| SP2-02-EFF1-230930-R12A | 32303357012 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32303357 Report date (mm/dd/yyyy): 10/12/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/13/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample SP3-01-T3-230926-R62 had elevated RLs most likely due to a lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 6/13/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32303593**

**10. Additional comments about lab report:** Twelve results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-231009-R66A | 32303593001 | PFBA |
| SP1-02-T1-231009-R66A | 32303593001 | PFOA |
| SP1-02-T1-231009-R66A | 32303593001 | PFOS |
| SP2-02-EFF1-231009-R66A | 32303593002 | PFBA |
| SP2-02-EFF1-231012-R67A | 32303593003 | PFBA |
| SP2-02-EFF1-231012-R67B | 32303593004 | PFBA |
| SP1-02-T1-231016-R68B | 32303593005 | PFBA |
| SP1-02-T1-231016-R68B | 32303593005 | PFOA |
| SP1-02-T1-231016-R68B | 32303593005 | PFOS |
| SP2-02-EFF1-231016-R68B | 32303593006 | PFBA |
| SP2-02-EFF1-231017-R69B | 32303593007 | PFBA |
| SP2-02-EFF1-231018-R70B | 32303593008 | PFBA |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32303593 Report date (mm/dd/yyyy): 10/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32303829**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ). Labeled analog exceedances and qualifications are summarized in the table below.

The acid labeled compound recoveries were low for the method blank and LCS. Most sample labeled compound recoveries were within criteria indicating the issue was isolated to the QC samples.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP2-02-EFF1-231030-R87B | 32303829002 | 13C2-PFDoA | 43.7 | UJ |
| SP2-02-EFF1-231101-R88B | 32303829004 | 13C2-PFDoA | 46.6 | UJ |
| SP1-02-T1-231103-R89A | 32303829005 | 13C2-PFDoA | 49.5 | UJ |
| SP2-02-EFF1-231103-R89A | 32303829006 | 13C2-PFDoA | 36.4 | UJ |
| SP2-02-EFF1-231103-R89A | 32303829006 | 13C7-PFuNA | 46.2 | UJ |
| SP1-02-T1-231107-R90B | 32303829007 | 13C2-PFDoA | 49.7 | UJ |
| SP2-02-EFF1-231107-R90B | 32303829008 | 13C2-PFDoA | 43.7 | UJ |

**10. Additional comments about lab report:** Sixteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-231030-R87B | 32303829001 | PFBA |
| SP1-02-T1-231030-R87B | 32303829001 | PFOA |
| SP1-02-T1-231030-R87B | 32303829001 | PFOS |
| SP2-02-EFF1-231030-R87B | 32303829002 | PFBA |
| SP1-02-T1-231101-R88B | 32303829003 | PFBA |
| SP1-02-T1-231101-R88B | 32303829003 | PFOA |
| SP1-02-T1-231101-R88B | 32303829003 | PFOS |
| SP2-02-EFF1-231101-R88B | 32303829004 | PFBA |
| SP1-02-T1-231103-R89A | 32303829005 | PFBA |
| SP1-02-T1-231103-R89A | 32303829005 | PFOA |
| SP1-02-T1-231103-R89A | 32303829005 | PFOS |
| SP2-02-EFF1-231103-R89A | 32303829006 | PFBA |
| SP1-02-T1-231107-R90B | 32303829007 | PFBA |
| SP1-02-T1-231107-R90B | 32303829007 | PFOA |
| SP1-02-T1-231107-R90B | 32303829007 | PFOS |
| SP2-02-EFF1-231107-R90B | 32303829008 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32303829 Report date (mm/dd/yyyy): 1/15/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided.<br>The case narrative indicates that the labeled standard recovery for 13C2-PFDoA was below control limits for CCV1. Results were qualified based on the 13C2-PFDoA recovery for each sample (see Section 4B). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32400288**

**10. Additional comments about lab report:** Four results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-240129-R90 | 32400288001 | PFBA |
| SP1-02-T1-240129-R90 | 32400288001 | PFOA |
| SP2-02-EFF1-240129-R90A | 32400288002 | PFBA |
| SP2-02-EFF1-240129-R90B | 32400288003 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32400288 Report date (mm/dd/yyyy): 2/2/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32400852**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated biased high (J+/UJ), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP1-02-T1-240314-R91A | 32400852001 | 13C4-PFBA | 44.5 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C5-PFPeA | 35.7 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C5-PFHxA | 31.1 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C4-PFHpA | 28.8 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C8-PFOA | 28.7 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C9-PFNA | 33.5 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C6-PFDA | 37.4 | J+ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C7-PFuNA | 40.7 | UJ |
| SP1-02-T1-240314-R91A | 32400852001 | 13C3-HFPO-DA | 27.3 | Not Reported |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C4-PFBA | 35.1 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C5-PFPeA | 27.8 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C5-PFHxA | 27.0 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C4-PFHpA | 26.7 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C8-PFOA | 29.1 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C9-PFNA | 34.1 | UJ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C3-HFPO-DA | 24.4 | Not Reported |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C6-PFDA | 39.4 | J+ |
| SP2-02-EFF1-240314-R91A | 32400852002 | 13C7-PFuNA | 45.4 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C4-PFBA | 43.6 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C5-PFPeA | 34.3 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C5-PFHxA | 29.3 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C4-PFHpA | 28.8 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C8-PFOA | 30.2 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C9-PFNA | 34.0 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C6-PFDA | 37.9 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C7-PFuNA | 43.3 | J+ |
| SP1-02-T1-240315-R92A | 32400852003 | 13C3-HFPO-DA | 28.5 | Not Reported |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C4-PFBA | 33.6 | J+ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C5-PFPeA | 27.5 | J+ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C5-PFHxA | 23.5 | J+ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C4-PFHpA | 22.3 | J+ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C8-PFOA | 23.0 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C9-PFNA | 26.0 | UJ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C6-PFDA | 33.3 | UJ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C7-PFuNA | 39.8 | UJ |
| SP2-02-EFF1-240315-R92A | 32400852004 | 13C3-HFPO-DA | 19.3 | Not Reported |
| SP1-02-T1-240321-R93A | 32400852005 | 13C4-PFBA | 38.2 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C5-PFPeA | 29.1 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C5-PFHxA | 26.0 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C4-PFHpA | 24.4 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C8-PFOA | 27.3 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C9-PFNA | 31.5 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C6-PFDA | 37.2 | J+ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C7-PFuNA | 42.7 | UJ |
| SP1-02-T1-240321-R93A | 32400852005 | 13C3-HFPO-DA | 22.4 | Not Reported |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C4-PFBA | 30.6 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C5-PFHxA | 24.1 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C5-PFPeA | 24.2 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C4-PFHpA | 26.2 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C5-PFHxA | 24.1 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C5-PFPeA | 24.2 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C8-PFOA | 30.6 | J+ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C9-PFNA | 36.7 | UJ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C6-PFDA | 42.4 | UJ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C7-PFuNA | 48.3 | UJ |
| SP2-02-EFF1-240321-R93A | 32400852006 | 13C3-HFPO-DA | 20.0 | Not Reported |

**10. Additional comments about lab report:** Sixteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-240314-R91A | 32400852001 | PFBA |
| SP1-02-T1-240314-R91A | 32400852001 | PFOA |
| SP1-02-T1-240314-R91A | 32400852001 | PFOS |
| SP2-02-EFF1-240314-R91A | 32400852002 | PFBA |
| SP1-02-T1-240315-R92A | 32400852003 | PFBA |
| SP1-02-T1-240315-R92A | 32400852003 | PFOA |
| SP1-02-T1-240315-R92A | 32400852003 | PFOS |
| SP2-02-EFF1-240315-R92A | 32400852004 | PFBA |
| SP1-02-T1-240321-R93A | 32400852005 | PFBA |
| SP1-02-T1-240321-R93A | 32400852005 | PFOA |
| SP1-02-T1-240321-R93A | 32400852005 | PFOS |
| SP2-02-EFF1-240321-R93A | 32400852006 | PFBA |
| SP1-02-T1-240305-R90A | 32400852009 | PFBA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-240305-R90A | 32400852009 | PFOA |
| SP1-02-T1-240305-R90A | 32400852009 | PFOS |
| SP2-02-EFF1-240305-R90A | 32400852010 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32400852 Report date (mm/dd/yyyy): 4/1/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☐ | ☒ | ☐ | Two samples were received outside on hold time and the analyses were cancelled. |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | The two samples that were received out side of hold time (and cancelled) are discussed in the case narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided.<br>The case narrative indicates that the labeled standard 13C2-PFDoA was above control limits in the bracketing CCV1 and CCV2. Associated results were nondetect and were acceptable without qualification. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32401050**

**10. Additional comments about lab report:** Sixteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| SP1-02-T1-240408-R17A | 32401050001 | PFBA |
| SP1-02-T1-240408-R17A | 32401050001 | PFOA |
| SP1-02-T1-240408-R17A | 32401050001 | PFOS |
| SP2-02-EFF1-240408-R17A | 32401050002 | PFBA |
| SP1-02-T1-240409-R14A | 32401050003 | PFBA |
| SP1-02-T1-240409-R14A | 32401050003 | PFOA |
| SP1-02-T1-240409-R14A | 32401050003 | PFOS |
| SP2-02-EFF1-240409-R14A | 32401050004 | PFBA |
| SP1-02-T1-240410-R15A | 32401050005 | PFBA |
| SP1-02-T1-240410-R15A | 32401050005 | PFOA |
| SP1-02-T1-240410-R15A | 32401050005 | PFOS |
| SP2-02-EFF1-240410-R15A | 32401050006 | PFBA |
| SP1-02-T1-240410-R16A | 32401050007 | PFBA |
| SP1-02-T1-240410-R16A | 32401050007 | PFOA |
| SP1-02-T1-240410-R16A | 32401050007 | PFOS |
| SP2-02-EFF1-240410-R16A | 32401050008 | PFBA |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32401050 Report date (mm/dd/yyyy): 4/17/2024

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86274 (WG85780)**

**Chain of custody preservation, and holding time (Section 1C):** The sample was received at a temperature of 7.3 °C, and was above the < 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C.

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85780-102 (A) | NFDHA | 132 | 70-130 | Associated results were nondetect, and were acceptable without qualification. |

**10. Additional comments about lab report:** For the results listed below, the peak was detected; however, it did not meet quantification criteria (ion ratios or signal to noise). These results were reported as a maximum possible concentration and qualified as estimated (J):

| Sample ID | Lab ID | Analyte |
|---|---|---|
| PZID-GW-22-27-01-041923 | L39228-90 R | PFOS |

Reporting limits (RLs) listed in the laboratory report were not included in the EQuIS EDD.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: P1007

Work order number/Lab report ID: DPWG86274 (WG85780) Report date (mm/dd/yyyy): 7/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 10/13/2023

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | The sample was received at a temperature of 7.3 °C, and was above the < 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Sample receipt temperature is discussed in the narrative. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used. ICAL and CCVs reviewed. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | Associated trip blanks were not reported in this SDG:<br>TRIP-COOLER8-042623 (L39228-8) |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank results are nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | Method limits of 50% to 150% used for samples. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances. Results did not require qualification. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD: RPDs ok |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± QL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☒ | ☐ | ☐ | |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2022).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 10/13/2023

# Attachment H-3
# AXYS Laboratory Validation Packages (DE-FLUORO™ Samples)

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: DPWG86050 (WG85602)**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ). Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| TRIP-COOLER1-230320 | L39012-1 | 13C4-PFBA | 49.3 | UJ |
| Batch1-T0-230228 | L39012-10 | d7-NMe-FOSE | 151 | No Qualifier |
| Batch1-T125-230324 | L39083-2 | d7-NMe-FOSE | 152 | No Qualifier |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Not all compounds reported were included in the OPRs (LCSs). Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS / LCSD %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| WG85602-102(A) / WG85602-103 | 4:2 FTS | 0 / 0 | 70 - 130 | Associated results were nondetect and were rejected (R) due to the severely low recoveries.<br><br>All samples were associated with this LCS/LCSD data. |
| | 6:2 FTS | 0.6 / 1.1 | 70 - 130 | |
| | 8:2 FTS | 1.3 / 0.9 | 70 - 130 | |
| | MeFOSAA | 0.1 / 0.2 | 70 - 130 | |
| | EtFOSAA | 0.1 / 0.1 | 70 - 130 | |

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300704**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below. Sample concentrations were greater than 10 times the blank concentration and results were acceptable without qualification.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 266903 | PFOA | 2.02 | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch 1-TO-230228 | 32300704001 | 13C2-4:2 FTS | 203 | Not Reported |
| Batch 1-TO-230228 | 32300704001 | 13C2-6:2 FTS | 207 | |
| Batch 1-TO-230228 | 32300704001 | 13C2-8:2 FTS | 205 | |
| Batch 1-TO-230228 | 32300704001 | 13C3-PFBS | 214 | J- |
| Batch 1-TO-230228 | 32300704001 | 13C3-PFHxS | 208 | J- |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-4:2 FTS | 192 | Not Reported |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-6:2 FTS | 199 | |
| Batch 1-TO(D)-230228 | 32300704002 | 13C2-8:2 FTS | 200 | |
| Batch 1-TO(D)-230228 | 32300704002 | 13C3-PFBS | 210 | J- |
| Batch 1-TO(D)-230228 | 32300704002 | 13C3-PFHxS | 203 | J- |
| Batch 1-T2-230228 | 32300704003 | 13C2-4:2 FTS | 196 | Not Reported |
| Batch 1-T2-230228 | 32300704003 | 13C2-6:2 FTS | 201 | |
| Batch 1-T2-230228 | 32300704003 | 13C2-8:2 FTS | 201 | |
| Batch 1-T2-230228 | 32300704003 | 13C3-PFBS | 210 | J- |
| Batch 1-T2-230228 | 32300704003 | 13C3-PFHxS | 207 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266904 | PFBA | 146 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFHxA | 132 | 70-130 | |
| | PFHpA | 139 | 70-130 | |
| | PFOA | 355 | 70-130 | |
| | PFBS | 134 | 70-130 | |
| | PFHxS | 131 | 70-130 | |
| | PFOS | 139 | 70-130 | |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 266904 | PFuNA | 132 | 70-130 | Associated results were nondetect and were acceptable without qualification. |
| | PFDoA | 132 | 70-130 | |

**10. Additional comments about lab report:** Twelve results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch 1-TO-230228 | 32300704001 | PFOA |
| Batch 1-TO-230228 | 32300704001 | PFHxS |
| Batch 1-TO-230228 | 32300704001 | PFOS |
| Batch 1-TO(D)-230228 | 32300704002 | PFOA |
| Batch 1-TO(D)-230228 | 32300704002 | PFHxS |
| Batch 1-TO(D)-230228 | 32300704002 | PFOS |
| Batch 1-T2-230228 | 32300704003 | PFOA |
| Batch 1-T2-230228 | 32300704003 | PFHxS |
| Batch 1-T2-230228 | 32300704003 | PFOS |
| Batch 1-T8-230301 | 32300704004 | PFOA |
| Batch 1-T8-230301 | 32300704004 | PFHxS |
| Batch 1-T8-230301 | 32300704004 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300704 Report date (mm/dd/yyyy): 3/7/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicate:<br>Batch 1-TO-230228 / Batch 1-TO(D)-230228 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples had elevated RLs due to sample arriving with lower sample size. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300720**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T24-230303 (50mL) | 32300720001 | 13C2-8:2 FTS | 154 | Not Reported |
| Batch1-T24-230303 (50mL) | 32300720001 | 13C3-PFBS | 162 | J- |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-4:2 FTS | 157 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-6:2 FTS | 168 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C2-8:2 FTS | 167 | Not Reported |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C3-PFBS | 169 | J- |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | 13C3-PFHxS | 154 | J- |

**10. Additional comments about lab report:** Eight results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T24-230303 (50mL) | 32300720001 | PFHpA |
| Batch1-T24-230303 (50mL) | 32300720001 | PFOA |
| Batch1-T24-230303 (50mL) | 32300720001 | PFHxS |
| Batch1-T24-230303 (50mL) | 32300720001 | PFOS |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFHpA |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFOA |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFHxS |
| Batch1-T24(D)-230303 (50mL) | 32300720003 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300720 Report date (mm/dd/yyyy): 3/8/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/2/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Two samples were listed on the Sample Summary, that were not reported and were not listed on the chain of custody (Batch1-T24-230303 (250mL) and Batch1-T24(D)-230303 |

| | | | | | |
|---|---|---|---|---|---|
| | | | | | (250mL)) |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☐ | ☒ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicates:<br>Batch1-T24-230303 (50mL) / Batch1-T24(D)-230303 (50mL) |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch1-T24-230303 (50mL) and Batch1-T24(D)-230303 (50mL) had elevated RLs due to sample arriving with lower sample size. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/2/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300755**

**Method blank detections (Section 3Bii):** Method blank detections are listed in the table below.

| Batch | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 267060 | PFOA | 2.38 | PFOA results were nondetect or greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). The laboratory indicated that the internal standard 13C4-PFOS was not detect due to severe matrix issues for samples Batch1-T28-Scrubber-Condensate and Batch1-T28-GAC-Condensate. Nineteen results were rejected (R) due to severely low labeled compound recoveries. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C2-PFDoA | 224 | J- |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-HFPO-DA | 34.9 | Not Reported |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-PFBS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C3-PFHxS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C4-PFHpA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C5-PFHxA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C6-PFDA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C7-PFuNA | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C8-PFOA | 3.7 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C8-PFOS | 0 | R |
| Batch1-T28-Scrubber-Condensate | 32300755002 | 13C9-PFNA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-4:2 FTS | 0 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C2-PFDoA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-HFPO-DA | 0 | Not Reported |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-PFBS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C3-PFHxS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C4-PFHpA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C5-PFHxA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C6-PFDA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C7-PFuNA | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C8-PFOA | 3.3 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C8-PFOS | 0 | R |
| Batch1-T28-GAC-Condensate | 32300755003 | 13C9-PFNA | 0 | R |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| Batch 267061 | PFOA | 232 | 70-130 | Associated detections were qualified as estimated biased high (J+), unless previously rejected (R) due to labeled analog recoveries.<br><br>Batch1-T28-Scrubber-Condensate<br>Batch1-T28-GAC-Condensate |
| | PFOS | 334 | 70-130 | |

**10. Additional comments about lab report:** Seventeen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to labeled analog recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T32-230307 | 32300755001 | PFHpA |
| Batch1-T32-230307 | 32300755001 | PFOA |
| Batch1-T32-230307 | 32300755001 | PFHxS |
| Batch1-T32-230307 | 32300755001 | PFOS |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFBA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHxA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHpA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFOA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFDA |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFHxS |
| Batch1-T28-Scrubber-Condensate | 32300755002 | PFOS |
| Batch1-T28-GAC-Condensate | 32300755003 | PFBA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHxA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T28-GAC-Condensate | 32300755003 | PFDA |
| Batch1-T28-GAC-Condensate | 32300755003 | PFHxS |
| Batch1-T28-GAC-Condensate | 32300755003 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300755 Report date (mm/dd/yyyy): 3/14/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections and discussion. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample Batch1-T32-230307 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Nineteen results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300882**

**10. Additional comments about lab report:** Six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T64-230310 | 32300882001 | PFHpA |
| Batch1-T64-230310 | 32300882001 | PFOA |
| Batch1-T64-230310 | 32300882001 | PFOS |
| Batch1-T64-230310 | 32300882001 | PFHxS |
| Batch1-T100-230320 | 32300882002 | PFOA |
| Batch1-T100-230320 | 32300882002 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300882 Report date (mm/dd/yyyy): 3/24/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch1-T64-230310 and Batch1-T100-230320 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32300960**

**10. Additional comments about lab report:** Two results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch1-T125-230324 | 32300960001 | PFOA |
| Batch1-T125-230324 | 32300960001 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32300960 Report date (mm/dd/yyyy): 3/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/10/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Sample Batch1-T125-230324 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301482**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that surrogate recoveries were low for samples Batch2-T8-230502 and Batch2-T24-230504 due to severe matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch2-T0-230501 | 32301482001 | 13C2-4:2 FTS | 162 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C2-6:2 FTS | 158 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C2-8:2 FTS | 160 | Not Reported |
| Batch2-T0-230501 | 32301482001 | 13C3-PFBS | 172 | J- |
| Batch2-T0-230501 | 32301482001 | 13C3-PFHxS | 165 | J- |
| Batch2-T2-230501 | 32301482002 | 13C2-4:2 FTS | 161 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C2-6:2 FTS | 156 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C2-8:2 FTS | 154 | Not Reported |
| Batch2-T2-230501 | 32301482002 | 13C3-PFBS | 176 | J- |
| Batch2-T2-230501 | 32301482002 | 13C3-PFHxS | 160 | J- |
| Batch2-T8-230502 | 32301482003 | 13C3-HFPO-DA | 30.4 | Not Reported |
| Batch2-T8-230502 | 32301482003 | 13C4-PFBA | 24.4 | J+ |
| Batch2-T8-230502 | 32301482003 | 13C5-PFPeA | 31.5 | J+ |
| Batch2-T8-230502 | 32301482003 | 13C5-PFHxA | 48.5 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C3-HFPO-DA | 46.2 | Not Reported |
| Batch2-T24-230504 | 32301482004 | 13C4-PFBA | 49.2 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C5-PFPeA | 47.9 | J+ |
| Batch2-T24-230504 | 32301482004 | 13C8-PFOA | 43.4 | J+ |

**10. Additional comments about lab report:** Thirteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T0-230501 | 32301482001 | PFOA |
| Batch2-T0-230501 | 32301482001 | PFHxS |
| Batch2-T0-230501 | 32301482001 | PFOS |
| Batch2-T2-230501 | 32301482002 | PFOA |
| Batch2-T2-230501 | 32301482002 | PFHxS |
| Batch2-T2-230501 | 32301482002 | PFOS |
| Batch2-T8-230502 | 32301482003 | PFOA |
| Batch2-T8-230502 | 32301482003 | PFHxS |
| Batch2-T8-230502 | 32301482003 | PFOS |
| Batch2-T24-230504 | 32301482004 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T24-230504 | 32301482004 | PFOA |
| Batch2-T24-230504 | 32301482004 | PFHxS |
| Batch2-T24-230504 | 32301482004 | PFOS |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301482 Report date (mm/dd/yyyy): 5/16/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301587**

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for samples Batch2-T41-230505 and Batch2-T125-GAC-Condensate-230 were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch2-T41-230505 | 32301587002 | 13C8-PFOA | 49.6 | J+ |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-4:2 FTS | 155 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-6:2 FTS | 160 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C2-8:2 FTS | 158 | Not Reported |
| Batch2-T125-Scrubber-Condensat | 32301587007 | 13C3-PFBS | 160 | J- |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C2-4:2 FTS | 267 | Not Reported |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C2-8:2 FTS | 42.7 | Not Reported |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C8-PFOA | 13.3 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C9-PFNA | 40.8 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C6-PFDA | 14.4 | J+ |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | 13C3-PFBS | 270 | J- |

**10. Additional comments about lab report:** Thirty-six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T41-230505 | 32301587002 | PFBA |
| Batch2-T41-230505 | 32301587002 | PFHxA |
| Batch2-T41-230505 | 32301587002 | PFHpA |
| Batch2-T41-230505 | 32301587002 | PFOA |
| Batch2-T41-230505 | 32301587002 | PFHxS |
| Batch2-T41-230505 | 32301587002 | PFOS |
| Batch2-T64-230506 | 32301587003 | PFHpA |
| Batch2-T64-230506 | 32301587003 | PFOA |
| Batch2-T64-230506 | 32301587003 | PFHxS |
| Batch2-T64-230506 | 32301587003 | PFOS |
| Batch2-T100-230508 | 32301587004 | PFBA |
| Batch2-T100-230508 | 32301587004 | PFHxA |
| Batch2-T100-230508 | 32301587004 | PFHpA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch2-T100-230508 | 32301587004 | PFOA |
| Batch2-T100-230508 | 32301587004 | PFHxS |
| Batch2-T100-230508 | 32301587004 | PFOS |
| Batch2-T125-230510 | 32301587005 | PFBA |
| Batch2-T125-230510 | 32301587005 | PFHxA |
| Batch2-T125-230510 | 32301587005 | PFHpA |
| Batch2-T125-230510 | 32301587005 | PFOA |
| Batch2-T125-230510 | 32301587005 | PFHxS |
| Batch2-T125-230510 | 32301587005 | PFOS |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFOA |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFHxS |
| Batch2-T125-Scrubber-230510 | 32301587006 | PFOS |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFHpA |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFOA |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFHxS |
| Batch2-T125-Scrubber-Condensat | 32301587007 | PFOS |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFBA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFHpA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFOA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFNA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFDA |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFHxS |
| Batch2-T125-GAC-Condensate-230 | 32301587008 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301587 Report date (mm/dd/yyyy): 5/23/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☐ | ☒ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☐ | ☒ | |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch2-T0-Scrubber-230501, Batch2-T64-230506, Batch2-T125-Scrubber-230510, Batch2-T125-Scrubber-Condensat, and Batch2-T125-GAC-Condensate-230 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for most samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301771**

**Chain of custody preservation, and holding time (Section 1C):** The samples were received at a temperature greater than the 6 °C criteria at 23.1°C. Associated detects associated were qualified as estimated biased low (J-), and the reporting limit for nondetects were qualified as estimated (UJ).

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the continuing calibration verification (CCV) bracketing the samples (6/2/2023 12:56) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Three results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances for 7 of the 8 samples were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to probable matrix interference for most samples. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-TO-230518 | 32301771001 | 13C2-4:2 FTS | 303 | Not Reported |
| Batch3-TO-230518 | 32301771001 | 13C2-6:2 FTS | 169 | Not Reported |
| Batch3-TO-230518 | 32301771001 | 13C8-PFOA | 28.3 | J+ |
| Batch3-TO-230518 | 32301771001 | 13C6-PFDA | 12.9 | J+ |
| Batch3-TO-230518 | 32301771001 | 13C3-PFBS | 311 | J- |
| Batch3-TO-230518 | 32301771001 | 13C3-PFHxS | 192 | J- |
| Batch3-TO(D)-230518 | 32301771002 | 13C2-4:2 FTS | 280 | Not Reported |
| Batch3-TO(D)-230518 | 32301771002 | 13C2-6:2 FTS | 161 | Not Reported |
| Batch3-TO(D)-230518 | 32301771002 | 13C8-PFOA | 29.4 | J+ |
| Batch3-TO(D)-230518 | 32301771002 | 13C6-PFDA | 13.8 | J+ |
| Batch3-TO(D)-230518 | 32301771002 | 13C3-PFBS | 291 | J- |
| Batch3-TO(D)-230518 | 32301771002 | 13C3-PFHxS | 187 | J- |
| Batch3-T2-230518 | 32301771003 | 13C2-4:2 FTS | 156 | Not Reported |
| Batch3-T2-230518 | 32301771003 | 13C2-8:2 FTS | 159 | Not Reported |
| Batch3-T2-230518 | 32301771003 | 13C2-PFDoA | 160 | J- |
| Batch3-T2-230518 | 32301771003 | 13C3-PFBS | 157 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T8-230518 | 32301771004 | 13C2-4:2 FTS | 196 | Not Reported |
| Batch3-T8-230518 | 32301771004 | 13C2-8:2 FTS | 178 | Not Reported |
| Batch3-T8-230518 | 32301771004 | 13C8-PFOA | 31.2 | J+ |
| Batch3-T8-230518 | 32301771004 | 13C6-PFDA | 35.1 | J+ |
| Batch3-T8-230518 | 32301771004 | 13C3-PFBS | 194 | J- |
| Batch3-T24-230522 | 32301771005 | 13C2-4:2 FTS | 229 | Not Reported |
| Batch3-T24-230522 | 32301771005 | 13C4-PFHpA | 32.1 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C8-PFOA | 11.0 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C9-PFNA | 25.9 | J+ |
| Batch3-T24-230522 | 32301771005 | 13C6-PFDA | 6.2 | R |
| Batch3-T24-230522 | 32301771005 | 13C3-PFBS | 260 | J- |
| Batch3-T24(D)-230522 | 32301771006 | 13C2-4:2 FTS | 294 | Not Reported |
| Batch3-T24(D)-230522 | 32301771006 | 13C4-PFHpA | 34.3 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C8-PFOA | 12.2 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C9-PFNA | 27;0 | J+ |
| Batch3-T24(D)-230522 | 32301771006 | 13C6-PFDA | 6.1 | R |
| Batch3-T24(D)-230522 | 32301771006 | 13C3-PFBS | 338 | J- |
| Batch3-T32-230522 | 32301771007 | 13C2-4:2 FTS | 186 | Not Reported |
| Batch3-T32-230522 | 32301771007 | 13C2-8:2 FTS | 37.1 | Not Reported |
| Batch3-T32-230522 | 32301771007 | 13C4-PFHpA | 31.2 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C8-PFOA | 10.6 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C9-PFNA | 22.5 | J+ |
| Batch3-T32-230522 | 32301771007 | 13C6-PFDA | 5.4 | R |
| Batch3-T32-230522 | 32301771007 | 13C3-PFBS | 212 | J- |
| Batch3-T32-230522 | 32301771007 | 13C8-PFOS | 48.8 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C2-8:2 FTS | 163 | Not Reported |
| Batch3-T66-230524 | 32301771008 | 13C4-PFHpA | 48.3 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C8-PFOA | 22.9 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C6-PFDA | 42.1 | J+ |
| Batch3-T66-230524 | 32301771008 | 13C2-PFDoA | 155 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOA | 222 | 70-130 | |
| | PFOS | 870 | 70-130 | |

**10. Additional comments about lab report:** Fifty-three results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-TO-230518 | 32301771001 | PFHpA |
| Batch3-TO-230518 | 32301771001 | PFOA |
| Batch3-TO-230518 | 32301771001 | PFNA |
| Batch3-TO-230518 | 32301771001 | PFDA |
| Batch3-TO-230518 | 32301771001 | PFHxS |
| Batch3-TO-230518 | 32301771001 | PFOS |
| Batch3-TO(D)-230518 | 32301771002 | PFHpA |
| Batch3-TO(D)-230518 | 32301771002 | PFOA |
| Batch3-TO(D)-230518 | 32301771002 | PFNA |
| Batch3-TO(D)-230518 | 32301771002 | PFDA |
| Batch3-TO(D)-230518 | 32301771002 | PFHxS |
| Batch3-TO(D)-230518 | 32301771002 | PFOS |
| Batch3-T2-230518 | 32301771003 | PFHpA |
| Batch3-T2-230518 | 32301771003 | PFOA |
| Batch3-T2-230518 | 32301771003 | PFHxS |
| Batch3-T2-230518 | 32301771003 | PFOS |
| Batch3-T8-230518 | 32301771004 | PFHpA |
| Batch3-T8-230518 | 32301771004 | PFOA |
| Batch3-T8-230518 | 32301771004 | PFNA |
| Batch3-T8-230518 | 32301771004 | PFHxS |
| Batch3-T8-230518 | 32301771004 | PFOS |
| Batch3-T24-230522 | 32301771005 | PFBA |
| Batch3-T24-230522 | 32301771005 | PFHxA |
| Batch3-T24-230522 | 32301771005 | PFHpA |
| Batch3-T24-230522 | 32301771005 | PFOA |
| Batch3-T24-230522 | 32301771005 | PFNA |
| Batch3-T24-230522 | 32301771005 | PFDA |
| Batch3-T24-230522 | 32301771005 | PFHxS |
| Batch3-T24-230522 | 32301771005 | PFOS |
| Batch3-T24(D)-230522 | 32301771006 | PFBA |
| Batch3-T24(D)-230522 | 32301771006 | PFHxA |
| Batch3-T24(D)-230522 | 32301771006 | PFHpA |
| Batch3-T24(D)-230522 | 32301771006 | PFOA |
| Batch3-T24(D)-230522 | 32301771006 | PFNA |
| Batch3-T24(D)-230522 | 32301771006 | PFDA |
| Batch3-T24(D)-230522 | 32301771006 | PFHxS |
| Batch3-T24(D)-230522 | 32301771006 | PFOS |
| Batch3-T32-230522 | 32301771007 | PFBA |
| Batch3-T32-230522 | 32301771007 | PFHxA |
| Batch3-T32-230522 | 32301771007 | PFHpA |
| Batch3-T32-230522 | 32301771007 | PFOA |
| Batch3-T32-230522 | 32301771007 | PFNA |
| Batch3-T32-230522 | 32301771007 | PFDA |
| Batch3-T32-230522 | 32301771007 | PFHxS |
| Batch3-T32-230522 | 32301771007 | PFOS |
| Batch3-T66-230524 | 32301771008 | PFBA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T66-230524 | 32301771008 | PFHxA |
| Batch3-T66-230524 | 32301771008 | PFHpA |
| Batch3-T66-230524 | 32301771008 | PFOA |
| Batch3-T66-230524 | 32301771008 | PFNA |
| Batch3-T66-230524 | 32301771008 | PFDA |
| Batch3-T66-230524 | 32301771008 | PFHxS |
| Batch3-T66-230524 | 32301771008 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

Results with both high and low bias are qualified as estimated (J), with no bias flag.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301771 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/21/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | The samples were received at a temperature of 23.1 °C and above the 6 °C criteria. Results for samples received at temperatures > 10 °C were qualfied as smmarized in the attached data qualification summary. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Field Duplicates:<br>Batch3-TO-230518 / Batch3-TO(D)-230518<br>Batch3-T24-230522 / Batch3-T24(D)-230522 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Three results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith
*(This document has been electronically signed.)*

Title: Environmental Chemist (CEAC)

Date (mm/dd/yyyy): 5/21/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301797**

**Calibration (Section 2A):** The case narrative indicated that PFOS recovery was high (135%) for the 6/2/2023 12:56 continuing calibration verification (CCV) due to carry over. The PFOS results were qualified as estimated biased high (J+).

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269543 (Batch HXX3474) | PFOS | 1.40 J | Associated sample concentrations were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Twelve results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to probable matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T98-230525 | 32301797001 | 13C2-6:2 FTS | 23.0 | Not Reported |
| Batch3-T98-230525 | 32301797001 | 13C2-8:2 FTS | 27.8 | Not Reported |
| Batch3-T98-230525 | 32301797001 | 13C5-PFHxA | 15.2 | J+ |
| Batch3-T98-230525 | 32301797001 | 13C4-PFHpA | 6.4 | R |
| Batch3-T98-230525 | 32301797001 | 13C6-PFDA | 5.8 | R |
| Batch3-T98-230525 | 32301797001 | 13C8-PFOA | 4.3 | R |
| Batch3-T98-230525 | 32301797001 | 13C9-PFNA | 5.9 | R |
| Batch3-T98-230525 | 32301797001 | 13C3-PFHxS | 30.6 | J+ |
| Batch3-T120-230526 | 32301797002 | 13C2-6:2 FTS | 29.1 | Not Reported |
| Batch3-T120-230526 | 32301797002 | 13C2-8:2 FTS | 27.2 | Not Reported |
| Batch3-T120-230526 | 32301797002 | 13C5-PFHxA | 16.3 | J+ |
| Batch3-T120-230526 | 32301797002 | 13C4-PFHpA | 5.9 | R |
| Batch3-T120-230526 | 32301797002 | 13C8-PFOA | 4.2 | R |
| Batch3-T120-230526 | 32301797002 | 13C9-PFNA | 5.7 | R |
| Batch3-T120-230526 | 32301797002 | 13C6-PFDA | 6.5 | R |
| Batch3-T120-230526 | 32301797002 | 13C3-PFBS | 251 | J- |
| Batch3-T120-230526 | 32301797002 | 13C3-PFHxS | 40.2 | J+ |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C2-4:2 FTS | 1560 | Not Reported |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C2-8:2 FTS | 21.9 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C5-PFHxA | 42.9 | J+ |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C4-PFHpA | 3.6 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C8-PFOA | 1.6 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C9-PFNA | 1.7 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C6-PFDA | 4.3 | R |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C3-PFBS | 1070 | J- |
| Batch3-T120-Scrubber-Condensat | 32301797003 | 13C3-PFHxS | 46.9 | J+ |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C2-4:2 FTS | 180 | Not Reported |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C8-PFOA | 31.1 | J+ |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C2-PFDoA | 154 | No Qualifier |
| Batch3-T120-Scrubber-230526 | 32301797004 | 13C3-PFBS | 187 | No Qualifier |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C2-4:2 FTS | 249 | Not Reported |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C8-PFOA | 13.6 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C9-PFNA | 43.5 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C6-PFDA | 21.3 | J+ |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | 13C3-PFBS | 268 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269544 (Batch HXX3474) | PFPeA | 131 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOA | 222 | 70-130 | |
| | PFOS | 870 | 70-130 | |

**10. Additional comments about lab report:** Eleven results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T98-230525 | 32301797001 | PFBA |
| Batch3-T98-230525 | 32301797001 | PFPeA |
| Batch3-T98-230525 | 32301797001 | PFHpA |
| Batch3-T98-230525 | 32301797001 | PFHxA |
| Batch3-T98-230525 | 32301797001 | PFOA |
| Batch3-T98-230525 | 32301797001 | PFNA |
| Batch3-T98-230525 | 32301797001 | PFDA |
| Batch3-T98-230525 | 32301797001 | PFHxS |
| Batch3-T98-230525 | 32301797001 | PFOS |
| Batch3-T120-230526 | 32301797002 | PFBA |
| Batch3-T120-230526 | 32301797002 | PFPeA |
| Batch3-T120-230526 | 32301797002 | PFHpA |
| Batch3-T120-230526 | 32301797002 | PFHxA |
| Batch3-T120-230526 | 32301797002 | PFOA |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch3-T120-230526 | 32301797002 | PFNA |
| Batch3-T120-230526 | 32301797002 | PFDA |
| Batch3-T120-230526 | 32301797002 | PFOS |
| Batch3-T120-230526 | 32301797002 | PFHxS |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFHpA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFOA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFNA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFDA |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFHxS |
| Batch3-T120-Scrubber-Condensat | 32301797003 | PFOS |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFHpA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFOA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFNA |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFHxS |
| Batch3-T120-Scrubber-230526 | 32301797004 | PFOS |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFHpA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFOA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFDA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFNA |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFHxS |
| Batch3-T120-GAC-Condensate-230 | 32301797005 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301797 Report date (mm/dd/yyyy): 6/6/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided (exceedances were discussed in the case narrative).<br>CCV exceedances and qualifications are discussed in the data qualifications summary (attachment). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Samples Batch3-T120-Scrubber-230526 and Batch3-T120-GAC-Condensate-230 had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for this sample, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Twelve results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32301998**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 269995 (Batch HXX3494) | PFOS | 3.43 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| 270009 (Batch HXX3496) | PFOA | 1.66 J | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 8.92 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). One result was rejected (R) due to a severely low labeled compound recovery. The case narrative indicated that the surrogate recovery exceedances for sample Batch4-T0-230612 were due to matrix interference, and also indicated that the internal standard 13C4-PFOS failed low due to matrix. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T0-230612 | 32301998001 | 13C2-4:2 FTS | 328 | Not Reported |
| Batch4-T0-230612 | 32301998001 | 13C2-6:2 FTS | 153 | Not Reported |
| Batch4-T0-230612 | 32301998001 | 13C8-PFOA | 22.5 | J+ |
| Batch4-T0-230612 | 32301998001 | 13C6-PFDA | 7.5 | R |
| Batch4-T0-230612 | 32301998001 | 13C3-PFBS | 339 | J- |
| Batch4-T0-230612 | 32301998001 | 13C3-PFHxS | 177 | J- |
| Batch4-T2-230614 | 32301998002 | 13C2-4:2 FTS | 230 | Not Reported |
| Batch4-T2-230614 | 32301998002 | 13C8-PFOA | 22.6 | J+ |
| Batch4-T2-230614 | 32301998002 | 13C6-PFDA | 17.6 | J+ |
| Batch4-T2-230614 | 32301998002 | 13C3-PFBS | 231 | J- |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances and qualifications are listed below. The case narrative indicates that the high recoveries are due to probable contamination from high level samples.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 269996 (Batch HXX3494) | PFOS | 135 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>Batch4-T0-230612<br>Batch4-T2-230614 |

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 270010 (Batch HXX3496) | PFOA | 275 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>Batch4-T1-Scrubber-230614 |
| | PFHxS | 136 | 70-130 | |
| | PFOS | 795 | 70-130 | |

**10. Additional comments about lab report:** Thirteen results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T0-230612 | 32301998001 | PFHxS |
| Batch4-T0-230612 | 32301998001 | PFHpA |
| Batch4-T0-230612 | 32301998001 | PFOA |
| Batch4-T0-230612 | 32301998001 | PFNA |
| Batch4-T0-230612 | 32301998001 | PFDA |
| Batch4-T0-230612 | 32301998001 | PFOS |
| Batch4-T2-230614 | 32301998002 | PFHpA |
| Batch4-T2-230614 | 32301998002 | PFOA |
| Batch4-T2-230614 | 32301998002 | PFNA |
| Batch4-T2-230614 | 32301998002 | PFDA |
| Batch4-T2-230614 | 32301998002 | PFHxS |
| Batch4-T2-230614 | 32301998002 | PFOS |
| Batch4-T1-Scrubber-230614 | 32301998003 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32301998 Report date (mm/dd/yyyy): 6/26/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 5/22/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volume. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**One result was rejected (R) due to a severely low labeled compound recovery.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 5/22/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302031**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270009 (Batch HXX3496) | PFOA | 1.66 J | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 8.92 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Two results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that the surrogate recovery exceedances were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T8-230615 | 32302031001 | 13C2-4:2 FTS | 166 | Not Reported |
| Batch4-T8-230615 | 32302031001 | 13C2-8:2 FTS | 181 | Not Reported |
| Batch4-T8-230615 | 32302031001 | 13C8-PFOA | 33.4 | J+ |
| Batch4-T8-230615 | 32302031001 | 13C3-PFBS | 169 | J- |
| Batch4-T26-230616 | 32302031002 | 13C2-6:2 FTS | 40.8 | Not Reported |
| Batch4-T26-230616 | 32302031002 | 13C3-HFPO-DA | 46.3 | Not Reported |
| Batch4-T26-230616 | 32302031002 | 13C5-PFHxA | 43.8 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C4-PFHpA | 25.8 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C8-PFOA | 7.9 | R |
| Batch4-T26-230616 | 32302031002 | 13C9-PFNA | 22.6 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C6-PFDA | 11.2 | J+ |
| Batch4-T26-230616 | 32302031002 | 13C3-PFHxS | 44.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C2-4:2 FTS | 168 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C2-6:2 FTS | 49.5 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C3-HFPO-DA | 45.9 | Not Reported |
| Batch4-T26(D)-230616 | 32302031003 | 13C5-PFHxA | 48.5 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C4-PFHpA | 25.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C8-PFOA | 9.0 | R |
| Batch4-T26(D)-230616 | 32302031003 | 13C9-PFNA | 20.2 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C6-PFDA | 11.4 | J+ |
| Batch4-T26(D)-230616 | 32302031003 | 13C3-PFBS | 198 | J- |
| Batch4-T32-230616 | 32302031004 | 13C4-PFHpA | 37.7 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T32-230616 | 32302031004 | 13C8-PFOA | 14.7 | J+ |
| Batch4-T32-230616 | 32302031004 | 13C6-PFDA | 23.6 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi and ii):** Recovery exceedances are listed below. The case narrative indicates that the high recoveries are due to probable contamination from high level samples.

| LCS IDs | Compound | LCS %Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| 270010 (Batch HXX3496) | PFOA | 275 | 70-130 | Associated detections were qualified as estimated biased high (J+):<br><br>All samples were associated with this LCS. |
| | PFOS | 795 | 70-130 | |
| | PFHxS | 136 | 70-130 | |

**10. Additional comments about lab report:** Thenty-six results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to severely low labeled compound recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T8-230615 | 32302031001 | PFHpA |
| Batch4-T8-230615 | 32302031001 | PFHxS |
| Batch4-T8-230615 | 32302031001 | PFOA |
| Batch4-T8-230615 | 32302031001 | PFOS |
| Batch4-T26-230616 | 32302031002 | PFBA |
| Batch4-T26-230616 | 32302031002 | PFHxA |
| Batch4-T26-230616 | 32302031002 | PFHpA |
| Batch4-T26-230616 | 32302031002 | PFOA |
| Batch4-T26-230616 | 32302031002 | PFNA |
| Batch4-T26-230616 | 32302031002 | PFHxS |
| Batch4-T26-230616 | 32302031002 | PFOS |
| Batch4-T26(D)-230616 | 32302031003 | PFBA |
| Batch4-T26(D)-230616 | 32302031003 | PFHxA |
| Batch4-T26(D)-230616 | 32302031003 | PFHpA |
| Batch4-T26(D)-230616 | 32302031003 | PFOA |
| Batch4-T26(D)-230616 | 32302031003 | PFNA |
| Batch4-T26(D)-230616 | 32302031003 | PFHxS |
| Batch4-T26(D)-230616 | 32302031003 | PFOS |
| Batch4-T32-230616 | 32302031004 | PFBA |
| Batch4-T32-230616 | 32302031004 | PFHxA |
| Batch4-T32-230616 | 32302031004 | PFHpA |
| Batch4-T32-230616 | 32302031004 | PFOA |
| Batch4-T32-230616 | 32302031004 | PFNA |
| Batch4-T32-230616 | 32302031004 | PFDA |
| Batch4-T32-230616 | 32302031004 | PFHxS |
| Batch4-T32-230616 | 32302031004 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302031 Report date (mm/dd/yyyy): 6/27/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/7/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 9.5 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Batch4-T26-230616 / Batch4-T26(D)-230616 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. **Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | All samples had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Two results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/7/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302108**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 270256 (Batch HXX/3507) | PFOA | 2.34 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |
| | PFOS | 2.66 | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results associated with recoveries less than 10% were rejected (R). Thirty-three results were rejected (R) due to severely low labeled compound recoveries. The case narrative indicated that most surrogate recovery exceedances were due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C2-4:2 FTS | 153 | Not Reported |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C2-8:2 FTS | 166 | Not Reported |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C8-PFOA | 49.3 | J+ |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C3-PFBS | 160 | J- |
| Batch4-T1-Scrubber-230612 | 32302108001 | 13C3-PFHxS | 153 | J- |
| Batch4-T64-230620 | 32302108002 | 13C2-4:2 FTS | 20.1 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C2-6:2 FTS | 13.5 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C2-8:2 FTS | 18.7 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C3-HFPO-DA | 5.7 | Not Reported |
| Batch4-T64-230620 | 32302108002 | 13C4-PFBA | 12.4 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C5-PFPeA | 25.6 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C5-PFHxA | 6.6 | R |
| Batch4-T64-230620 | 32302108002 | 13C4-PFHpA | 4.4 | R |
| Batch4-T64-230620 | 32302108002 | 13C8-PFOA | 1.9 | R |
| Batch4-T64-230620 | 32302108002 | 13C9-PFNA | 3.0 | R |
| Batch4-T64-230620 | 32302108002 | 13C6-PFDA | 3.0 | R |
| Batch4-T64-230620 | 32302108002 | 13C7-PFuNA | 20.5 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C2-PFDoA | 30.6 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C3-PFBS | 41.1 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C3-PFHxS | 16.4 | J+ |
| Batch4-T64-230620 | 32302108002 | 13C8-PFOS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C2-4:2 FTS | 0 | Not Reported |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T102-230622 | 32302108003 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C3-HFPO-DA | 9.4 | Not Reported |
| Batch4-T102-230622 | 32302108003 | 13C4-PFBA | 31.2 | J+ |
| Batch4-T102-230622 | 32302108003 | 13C4-PFHpA | 3.6 | R |
| Batch4-T102-230622 | 32302108003 | 13C5-PFHxA | 12.6 | J+ |
| Batch4-T102-230622 | 32302108003 | 13C8-PFOA | 2.1 | R |
| Batch4-T102-230622 | 32302108003 | 13C9-PFNA | 1.8 | R |
| Batch4-T102-230622 | 32302108003 | 13C6-PFDA | 5.0 | R |
| Batch4-T102-230622 | 32302108003 | 13C7-PFuNA | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C2-PFDoA | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C8-PFOS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C3-PFBS | 0 | R |
| Batch4-T102-230622 | 32302108003 | 13C3-PFHxS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C3-HFPO-DA | 5.4 | Not Reported |
| Batch4-T125-230623 | 32302108004 | 13C4-PFBA | 16.5 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C5-PFPeA | 49.9 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C5-PFHxA | 10.3 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C4-PFHpA | 4.4 | R |
| Batch4-T125-230623 | 32302108004 | 13C8-PFOA | 1.4 | R |
| Batch4-T125-230623 | 32302108004 | 13C9-PFNA | 1.2 | R |
| Batch4-T125-230623 | 32302108004 | 13C6-PFDA | 2.3 | R |
| Batch4-T125-230623 | 32302108004 | 13C7-PFuNA | 44.8 | J+ |
| Batch4-T125-230623 | 32302108004 | 13C2-PFDoA | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C3-PFBS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C3-PFHxS | 0 | R |
| Batch4-T125-230623 | 32302108004 | 13C8-PFOS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-6:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-8:2 FTS | 0 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-HFPO-DA | 263 | Not Reported |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C5-PFHxA | 271 | J- |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C4-PFHpA | 46.4 | J+ |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C8-PFOA | 5.2 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C9-PFNA | 2.4 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C6-PFDA | 12.2 | J+ |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C7-PFuNA | 158 | J- |

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C2-PFDoA | 249 | J- |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-PFBS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C3-PFHxS | 0 | R |
| Batch4-T125-Scrubber-Condensat | 32302108005 | 13C8-PFOS | 0 | R |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C2-4:2 FTS | 157 | Not Reported |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C2-8:2 FTS | 182 | Not Reported |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C3-PFBS | 174 | No Qualifier |
| Batch4-T125-Scrubber-230623 | 32302108006 | 13C3-PFHxS | 189 | J- |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C2-4:2 FTS | 0 | Not Reported |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C2-8:2 FTS | 19.7 | Not Reported |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C4-PFHpA | 20.7 | J+ |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C8-PFOA | 5.3 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C9-PFNA | 3.6 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C6-PFDA | 5.3 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C3-PFBS | 0 | R |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | 13C8-PFOS | 0 | R |

**10. Additional comments about lab report:** Forty-nine results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J), unless previously rejected (R) due to severely low labeled compound recoveries.

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T1-Scrubber-230612 | 32302108001 | PFOA |
| Batch4-T1-Scrubber-230612 | 32302108001 | PFHxS |
| Batch4-T1-Scrubber-230612 | 32302108001 | PFOS |
| Batch4-T64-230620 | 32302108002 | PFBA |
| Batch4-T64-230620 | 32302108002 | PFPeA |
| Batch4-T64-230620 | 32302108002 | PFHpA |
| Batch4-T64-230620 | 32302108002 | PFHxA |
| Batch4-T64-230620 | 32302108002 | PFOA |
| Batch4-T64-230620 | 32302108002 | PFNA |
| Batch4-T64-230620 | 32302108002 | PFDA |
| Batch4-T64-230620 | 32302108002 | PFHxS |
| Batch4-T64-230620 | 32302108002 | PFOS |
| Batch4-T102-230622 | 32302108003 | PFBA |
| Batch4-T102-230622 | 32302108003 | PFPeA |
| Batch4-T102-230622 | 32302108003 | PFHxA |
| Batch4-T102-230622 | 32302108003 | PFHpA |
| Batch4-T102-230622 | 32302108003 | PFOA |
| Batch4-T102-230622 | 32302108003 | PFNA |
| Batch4-T102-230622 | 32302108003 | PFDA |
| Batch4-T102-230622 | 32302108003 | PFHxS |
| Batch4-T102-230622 | 32302108003 | PFOS |

| Field ID | Lab ID | Analyte |
|---|---|---|
| Batch4-T125-230623 | 32302108004 | PFBA |
| Batch4-T125-230623 | 32302108004 | PFPeA |
| Batch4-T125-230623 | 32302108004 | PFHxA |
| Batch4-T125-230623 | 32302108004 | PFHpA |
| Batch4-T125-230623 | 32302108004 | PFNA |
| Batch4-T125-230623 | 32302108004 | PFOA |
| Batch4-T125-230623 | 32302108004 | PFDA |
| Batch4-T125-230623 | 32302108004 | PFHxS |
| Batch4-T125-230623 | 32302108004 | PFOS |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFHpA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFOA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFNA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFDA |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFHxS |
| Batch4-T125-Scrubber-Condensat | 32302108005 | PFOS |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFOA |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFDA |
| Batch4-T125-Scrubber-230623 | 32302108006 | PFOS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFBA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFPeA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHxA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHpA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFOA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFNA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFDA |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFBS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFHxS |
| Batch4-T125-GAC-Condensate-230 | 32302108007 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302108 Report date (mm/dd/yyyy): 6/30/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/7/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 8.8 °C, and above the 6 °C criteria. Results were not qualified, as the sample was received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | Calibration data not provided.<br>The case narrative indicated that the PFOS CCV recovery was above limits for CCV2, and that low level concentrations may be biased high. PFOS sample concentrations exceeded the instrument calibration range, and were qualified as estimated (J). |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Three samples (Batch4-T1-Scrubber-230612, Batch4-T125-Scrubber-230623, and Batch4-T125-GAC-Condensate-230) had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**Thirty-three results were rejected (R) due to severely low labeled compound recoveries.**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/10/2024

**Laboratory Data Review Checklist Attachment**
**Data Validation Qualifiers**
**Project: MPCA P1007**
**AXYS SDG: 32302437**

**Method blank detections (Section 3Aii):** Method blank detections are listed in the table below.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified |
|---|---|---|---|
| 271211 (Batch XLC2448) | PFOS | 7.39 | Associated sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with high recoveries were qualified as estimated biased low (J-) and results associated with low recoveries were qualified as estimated biased high (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. The case narrative indicated that the surrogate recovery exceedances for samples DRUM1-PIKE-RINSE-METHANOL-0720 and DRUM3-PIKE-RINSE-WATER-072023 were due to matrix interference; and that the internal standard 13C4-PFOS was outside control limits due to matrix interference. Labeled analog exceedances and qualifications are summarized in the table below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (limits 50% to 150%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-4:2 FTS | 263 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-6:2 FTS | 247 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C2-8:2 FTS | 201 | Not Reported |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C6-PFDA | 29.6 | J+ |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C3-PFBS | 283 | No Qualifier |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | 13C3-PFHxS | 272 | J- |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-4:2 FTS | 181 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-6:2 FTS | 167 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C2-8:2 FTS | 152 | Not Reported |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C3-PFBS | 195 | No Qualifier |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | 13C3-PFHxS | 187 | J- |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-HFPO-DA | 45.4 | Not Reported |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C5-PFPeA | 44.2 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C4-PFHpA | 48.9 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C8-PFOA | 45.6 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C9-PFNA | 48.1 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C6-PFDA | 41.4 | J+ |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-PFBS | 160 | No Qualifier |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | 13C3-PFHxS | 157 | J- |

**10. Additional comments about lab report:** Eight results were quantitated above the instrument calibration range as listed below, and are qualified as estimated (J).

| Field ID | Lab ID | Analyte |
|---|---|---|
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFOA |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFDA |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFHxS |
| DRUM1-PIKE-RINSE-METHANOL-0720 | 32302437001 | PFOS |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | PFOA |
| DRUM2-PIKE-RINSE-SULFURIC-0720 | 32302437002 | PFOS |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | PFOA |
| DRUM3-PIKE-RINSE-WATER-072023 | 32302437003 | PFOS |

Results qualified as estimated (J) by the laboratory for detections below the LOQ/CL are accepted by validator.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: 32302437 Report date (mm/dd/yyyy): 8/3/2023

Laboratory: SGS AXYS Review date (mm/dd/yyyy): 6/12/2024

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Samples were received at a temperature of 7.1 °C, and above the 6 °C criteria. Results were not qualified, as the samples were received at temperatures < 10 °C. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☐ | ☒ | Calibration data not provided. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualifications summary (attachment) for method blank detections. Results did not require qualification. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☐ | ☒ | |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits (100±50%) used for review. |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualifications summary (attachment) for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☐ | ☐ | ☒ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☐ | ☒ | ☐ | |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☐ | ☒ | 30% RPD limit, or ± 2xRL, used for water matrix. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | The samples had elevated RLs due to lower sample volumes. However, analytes exceeded the instrument calibration for the samples, so it is unlikely that lower RLs are achievable. |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (October 2023).**

**Qualifications for results quantitated above the instrument calibration range are discussed in the qualifications summary (attachment).**

**See attachment for additional comments and qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 6/12/2024

# Attachment H-4
# ALS Laboratory Validation Packages

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008566 Report date (mm/dd/yyyy): 10/8/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/22/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but not listed on the COC (results were not reported for the trip blank in this SDG - see SDG K2008577).<br>COCs listed many water samples that are not reported in this SDG. Receipt form discusses issues with water samples (2 listed on COC were not received, and two additional samples were received that were not listed on COC). |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used during review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported in K2008577 |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank was nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL:<br>PFOA 0.16 J ng/g |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for associated samples and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS sed/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | Laboratory duplicate |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | Laboratory duplicate<br>FDs: RC22-SED-0-6-01-091820/RC22-SED-0-6-02-091820 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 50% RPD limit for PFAS (sed). RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/22/2020

## Data Validation Qualifications for K2008566

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were qualified as estimated (UJ and J):<br><br>RC7-SED-0-6-01-092120 (K2008566-001)<br>EP19-SED-0-6-01-092020 (K2008566-005)<br>EP27C-SED-0-6-01-091520 (K2008566-006)<br>EP26A-SED-0-6-01-091720 (K2008566-007)<br>RC16A-SED-0-6-01-092120 (K2008566-008)<br>RC18-SED-0-6-01-091720 (K2008566-009)<br>WL5-SED-0-6-01-091720 (K2008566-011)<br>RC7-SED-0-6-01-092020 (K2008566-013) |
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>All samples were associated with this ICV. |
| CCV KC2000531 (10/6/2020) 698188 | PFOA | 151% | 70-130% | No results reported from this CCV. |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| Method Blank KQ2014193-05 | PFOA | 0.16 J | RC7-SED-0-6-01-092120 (K2008566-001)<br>RC3A-SED-0-6-01-092020 (K2008566-002)<br>RC4A-SED-0-6-01-091820 (K2008566-003)<br>RC5A-SED-0-6-01-091820 (K2008566-004)<br>EP19-SED-0-6-01-092020 (K2008566-005)<br>EP26A-SED-0-6-01-091720 (K2008566-007)<br>RC16A-SED-0-6-01-092120 (K2008566-008)<br>RC18-SED-0-6-01-091720 (K2008566-009)<br>WL5-SED-0-6-01-091720 (K2008566-011)<br>RC7-SED-0-6-01-092020 (K2008566-013)<br>RC22-SED-0-6-01-091820 (K2008566-014) |
| CCB 9/30/20 20:25 | PFPeA | 0.31 J | RC7-SED-0-6-01-092120 (K2008566-001)<br>RC5A-SED-0-6-01-091820 (K2008566-004)<br>EP27C-SED-0-6-01-091520 (K2008566-006)<br>RC6A-SED-0-6-02-092120091820 (K2008566-015) |
| | PFOA | 0.25 J | RC7-SED-0-6-01-092120 (K2008566-001)<br>RC3A-SED-0-6-01-092020 (K2008566-002)<br>RC4A-SED-0-6-01-091820 (K2008566-003)<br>RC5A-SED-0-6-01-091820 (K2008566-004)<br>EP19-SED-0-6-01-092020 (K2008566-005)<br>EP26A-SED-0-6-01-091720 (K2008566-007)<br>RC16A-SED-0-6-01-092120 (K2008566-008)<br>RC18-SED-0-6-01-091720 (K2008566-009) |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| | | | WL5-SED-0-6-01-091720 (K2008566-011)<br>RC7-SED-0-6-01-092020 (K2008566-013)<br>RC22-SED-0-6-01-091820 (K2008566-014) |
| | PFNA | 0.36 J | RC3A-SED-0-6-01-092020 (K2008566-002)<br>RC4A-SED-0-6-01-091820 (K2008566-003)<br>EP27C-SED-0-6-01-091520 (K2008566-006)<br>RC6A-SED-0-6-01-092120 (K2008566-012)<br>RC22-SED-0-6-01-091820 (K2008566-014) |
| | PFDA | 0.27 J | EP1A-SED-0-6-01-091520 (K2008566-010)<br>EP27C-SED-0-6-01-091520 (K2008566-006)<br>RC6A-SED-0-6-01-092120 (K2008566-012)<br>RC22-SED-0-6-01-091820 (K2008566-014)<br>RC6A-SED-0-6-02-092120 (K2008566-015) |
| | PFTeDA | 0.29 J | RC3A-SED-0-6-01-092020 (K2008566-002)<br>RC4A-SED-0-6-01-091820 (K2008566-003)<br>RC5A-SED-0-6-01-091820 (K2008566-004)<br>EP27C-SED-0-6-01-091520 (K2008566-006)<br>EP26A-SED-0-6-01-091720 (K2008566-007)<br>RC16A-SED-0-6-01-092120 (K2008566-008)<br>RC18-SED-0-6-01-091720 (K2008566-009)<br>EP1A-SED-0-6-01-091520 (K2008566-010)<br>WL5-SED-0-6-01-091720 (K2008566-011)<br>RC7-SED-0-6-01-092020 (K2008566-013) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008577 Report date (mm/dd/yyyy): 10/27/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/22/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | Sample WL5-POWAT-01-091720 (K2008577-006) was re-extracted 21 days over hold time. See data qualification summary for qualifications. |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☐ | ☒ | ☐ | No sample summary table included in the report. |

| | | | | | |
|---|---|---|---|---|---|
| | | | | | The lab report for the long list of PFAS analytes lists the field ID for sample K2008577-005 as B3-WAT-BULK-02-091620 rather than what is on the COC (B3-WAT-BULK-01-091620). |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank was nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Two results rejected due to severly low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>KQ2014877-01/KQ2014877-02:<br>PFHpS 41% RPD<br>The associated result was nondetect. No qualifiers. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Two results rejected due to severly low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/23/2020

## Data Validation Qualifications for K2008577

**Holding Times (Section 1F):** Sample WL5-POWAT-01-091720 (K2008577-006) was re-extracted 21 days over hold time. Associated results (EtFOSA, EtFOSAA, MeFOSAA, PFDS, and PFTrDA) were nondetect and qualified UJ.

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV K2008577 (10/06/20 19:30) 698188 | PFOA | 151 %R | 70-130% | Results not reported from this CCV. |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | J+: BPI-WAT-MID-01-091920 |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | |
| | PFTeDA | 170 %R | 70-130% | |
| | NMeFOSE | 267 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | NEtFOSE | 292 %R | 70-130% | |
| | 6:2 FTS | 134 %R | 70-130% | |
| CCV KC2000537 (10/07/20 05:53) 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000572 (10/23/20 5:52) 700485 | PFTeDA | 142 %R | 70-130% | Results not reported from this CCV. |
| | EtFOSA | 143 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | NEtFOSE | 185 %R | 70-130% | |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014290-03 | 9/29/2020 15:17 | MeFOSA | 0.51 J | Associated results were nondetect. |
| | | PFDoDA | 1.4 J | WL5-POWAT-01-091720 (K2008577-006) |
| | | PFTeDA | 2.6 J | WL5-POWAT-01-091720 (K2008577-006) VB3-WAT-BULK-02-091620 (K2008577-005) |
| | | PFTrDA | 3.6 J | |
| KQ2014429-03 | 10/1/2020 7:40 | MeFOSA | 0.75 J | BPI-WAT-MID-01-091920 (K2008577-003) |
| | | PFTeDA | 3.7 J | |
| | | PFBS | 0.3 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | N-EtFOSE | 0.93 J | |
| | | N-MeFOSE | 1.1 J | |
| KQ2014877-03 | 10/7/2020 7:56 | PFBA | 0.53 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| KQ2016108-03 | 10/22/2020 8:46 | N-EtFOSE | 0.18 J | No associated results. |
| | | MeFOSA | 0.62 J | |
| | | PFOA | 0.99 J | |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | EP24-WAT-BULK-01-091620 (K2008577-004) |
| | | EtFOSA | 0.40 J | Associated results were nondetect. |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | BPI-WAT-MID-01-091920 (K2008577-003) |
| | | PFTeDA | 3.1 J | |
| | | MeFOSA | 0.58 J | |
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | RC21-WAT-BULK-01-092120 (K2008577-007)<br>RC14-WAT-BULK-01-092120 (K2008577-009)<br>RC13-WAT-BULK-01-092120 (K2008577-011)<br>RC18A-WAT-BULK-01-092120 (K2008577-013) |
| | | MeFOSA | 0.62 J | Associated results were nondetect. |
| | | EtFOSAA | 0.92 J | WL10-WAT-BULK-01-092120 (K2008577-016) |
| KQ2015034-02 | 10/8/20 21:12 | MeFOSA | 0.63 J | Associated results were nondetect. |
| | | EtFOSA | 0.38 J | |
| | | N-EtFOSE | 0.18 J | |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | Associated results were nondetect. |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | No associated results. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| BPI-WAT-MID-02-091920 | K2008577-001 | 13C2-4:2 FTS | 248 | No qualifier |
| | | 13C2-PFTeDA | 40 | UJ |
| BPI-WAT-MID-01-091920 | K2008577-002 | 13C2-4:2 FTS | 305 | No qualifier |
| | | 13C2-6:2 FTS | 250 | No qualifier |
| | | 13C2-PFTeDA | 49 | UJ |
| BPI-WAT-MID-01-091920 | K2008577-003 | 13C2-4:2 FTS | 246 | No qualifier |
| | | D9-EtFOSE | 37 | UJ |
| EP24-WAT-BULK-01-091620 | K2008577-004 | 13C2-4:2 FTS | 274 | No qualifier |
| | | 13C2-6:2 FTS | 201 | No qualifier |
| VB3-WAT-BULK-02-091620 | K2008577-005 | 13C2-PFDoDA | 44 | UJ |
| | | 13C2-PFTeDA | 5 | R |
| | | D3-MeFOSA | 40 | UJ |
| | | D3-MeFOSAA | 36 | UJ |
| | | D5-EfFOSAA | 33 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | D5-EtFOSA | 27 | UJ |
| | | D7-MeFOSE | 42 | UJ |
| | | D9-EtFOSE | 25 | UJ |
| WL5-POWAT-01-091720 | K2008577-006 | 13C2-PFDoDA | 37 | J+ |
| | | 13C2-PFTeDA | 3 | R |
| | | D3-MeFOSA | 46 | UJ |
| | | D3-MeFOSAA | 31 | UJ |
| | | D5-EfFOSAA | 25 | UJ |
| | | D7-MeFOSE | 44 | UJ |
| | | D9-EtFOSE | 30 | UJ |
| RC21-WAT-BULK-01-092120 | K2008577-007 | 13C2-4:2 FTS | 238 | No qualifier |
| | | 13C2-6:2 FTS | 212 | No qualifier |
| | | 13C2-PFTeDA | 33 | UJ |
| RC21-WAT-BULK-01-092120 | K2008577-008 | 13C2-PFTeDA | 45 | UJ |
| RC14-WAT-BULK-01-092120 | K2008577-009 | 13C2-4:2 FTS | 251 | No qualifier |
| | | 13C2-6:2 FTS | 249 | No qualifier |
| | | 13C2-PFTeDA | 36 | UJ |
| RC14-WAT-BULK-01-092120 | K2008577-010 | 13C2-PFTeDA | 45 | UJ |
| RC13-WAT-BULK-01-092120 | K2008577-011 | 13C2-4:2 FTS | 288 | No qualifier |
| | | 13C2-6:2 FTS | 232 | No qualifier |
| | | 13C2-PFTeDA | 37 | UJ |
| RC13-WAT-BULK-01-092120 | K2008577-012 | 13C2-PFTeDA | 45 | UJ |
| RC18A-WAT-BULK-01-092120 | K2008577-013 | 13C2-4:2 FTS | 222 | No qualifier |
| | | 13C2-6:2 FTS | 220 | No qualifier |
| | | 13C2-PFTeDA | 45 | UJ |
| WL10-WAT-BULK-01-092120 | K2008577-017 | 13C2-PFTeDA | 44 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) and detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014290-01/ KQ2014290-02 (698190) | 11Cl-PF3OUdS | 73/**68** | 70-130 | Associated results were nondetect and qualified UJ:<br><br>VB3-WAT-BULK-02-091620 (K2008577-005)<br>WL5-POWAT-01-091720 (K2008577-006) |
| KQ2014290-01/ KQ2014290-02 (699134) | MeFOSAA | 122/**132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoDA | **146/133** | 70-130 | J+:<br>WL5-POWAT-01-091720 (K2008577-006) |
| | PFUnDA | 126/**131** | 70-130 | |
| | PFPeS | **143/**125 | 70-130 | J+:<br>VB3-WAT-BULK-02-091620 (K2008577-005)<br>WL5-POWAT-01-091720 (K2008577-006) |

| LCS/LCSD IDs (Batch) | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014290-01/ KQ2014290-02 (699503) | PFDS | **67/58** | 70-130 | Associated results were nondetect and qualified UJ:<br><br>VB3-WAT-BULK-02-091620 (K2008577-005)<br>WL5-POWAT-01-091720 (K2008577-006) |
| | PFTrDA | **148/157** | 70-130 | J+:<br>VB3-WAT-BULK-02-091620 (K2008577-005)<br>WL5-POWAT-01-091720 (K2008577-006) |
| KQ2014429-01/ KQ2014429-02 (698189) | 4:2 FTS | **137/**120 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **138/**125 | 70-130 | |
| | PFBA | **134/**130 | 70-130 | J+:<br>BPI-WAT-MID-01-091920 (K2008577-003) |
| KQ2014429-01/ KQ2014429-02 (699503) | PFPeS | **166/153** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFTrDA | **139/**109 | 70-130 | J+:<br>BPI-WAT-MID-01-091920 (K2008577-003) |
| KQ2016108-01/ KQ2016108-02 (700485) | EtFOSA | **154/145** | 70-130 | Associated results were nondetect. No qualifiers. |
| | NEtFOSE | **187/226** | 70-130 | |
| | NMeFOSE | **137/153** | 70-130 | |
| | PFTeDA | **134/**124 | 70-130 | J+:<br>WL5-POWAT-01-091720 (K2008577-006) |
| KQ2014635-01/ KQ2014635-02 | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoA | 129/**149** | 70-130 | |
| | PFHpS | **132/136** | 70-130 | J+:<br>WL10-WAT-BULK-01-092120 (K2008577-017) |
| | PFPeS | **146/141** | 70-130 | J+:<br>RC17-POWAT-01-092120 (K2008577-015)<br>WL10-WAT-BULK-01-092120 (K2008577-017) |
| | PFOS | 127/**132** | 70-130 | J+:<br>RC21-WAT-BULK-01-092120 (K2008577-008)<br>RC14-WAT-BULK-01-092120 (K2008577-010)<br>RC13-WAT-BULK-01-092120 (K2008577-012)<br>RC18A-WAT-BULK-01-092120 (K2008577-014)<br>RC17-POWAT-01-092120 (K2008577-015)<br>WL10-WAT-BULK-01-092120 (K2008577-017) |
| KQ2014877-01/ KQ2014877-02 | 11Cl-PF3OUdS | **138/147** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFHxS | 119/**137** | 70-130 | |
| | PFNS | **136/139** | 70-130 | |
| | PFPeS | **135/**114 | 70-130 | |
| KQ2014524-01/ KQ2014524-01 (698467) | PFPeS | 130/**133** | 70-130 | J+:<br>RC13-WAT-BULK-01-092120-U (K2008577-011)<br>RC18A-WAT-BULK-01-092120-U (K2008577-013) |

**ALS K2008578** 11/22/20 **LS**

Initial Cal 9/29/20 **PFOS**

| Area Counts ICAL | | | |
|---|---|---|---|
| IS (M8PFOS) | PFOS stds (cal form) | Conc (ng/mL) | RFF - Cal form (p 229) |
| Area Counts not reported for ICAL | | 0.0465 | 1.0330 |
| | | 0.0929 | 0.9826 |
| | | 0.4646 | 0.8663 |
| | | 0.9292 | 0.9093 |
| | | 4.6461 | 0.8484 |
| | | 9.2923 | 0.9116 |
| | | 13.9385 | 0.9536 |

PFOS Std chk:

| | | |
|---|---|---|
| 0.929229157 | 50 | 46.46145785 |

AverageCalc: 0.92926 (0.9293 reported)

RSD Calc: 7.00340 (7.0 report)

| Standard | Area | IS | Conc CCV calc ng/ml | Conc CCV reported ng/ml | True Value ng/L | %R Calculated | %D Calc | Reported | |
|---|---|---|---|---|---|---|---|---|---|
| ICV 09/29/2020 21:07 | Not Reported | Not Reported | #VALUE! | 0.851 | 0.9290 | 91.60 | -8.40% | -8.389 %D | p. 264 |
| CCV 10/1/20 18:00 | 160921 | 1092527 | 0.793 | 0.7924 | 0.9290 | 85.30 | -- | 85.3 %R | p. 272, 2050 |

| Sample | Compound | Area | Int Std | Dil Factor | Calculated Extract ng/ml | Reported Extract ng/ml | Calculated ng/L | Reported ng/L | Intial volume (L) | Final Volume (L) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| RC3-WAT-BULK-01-091620 (-001) | PFOS | 809370 | 77607 | 10 | 56.115 | 56.108 | 1481.40 | 1500 | 0.303 | 0.008 | 10/2/2020 19:21 | p. 109, 746 |
| RC3-WAT-BULK-03-091620 (-012) | PFOS | 788540 | 93011 | 10 | 45.617 | 45.6108 | 1280.30 | 1300 | 0.285 | 0.008 | 10/2/2020 19:42 | p. 131, 1116 |
| LCS 10/1/20 697611 | PFOS | 153391 | 1201882 | 1 | 0.6867 | 0.6866 | 21.97 | 22 | 0.25 | 0.008 | 10/1/2020 18:21 | p. 181, 1380 |
| MS (-012) | PFOS | 6532518 | 848392 | 1 | 41.4303 | 41.4249 | 1142.76 | 1140 E | 0.29 | 0.008 | 10/1/2020 19:15 | p. 179, 1791 |
| MSD (-012) | PFOS | 5410089 | 640065 | 1 | 45.4794 | 45.4735 | 1299.24 | 1300 E | 0.28 | 0.008 | 10/1/2020 19:25 | p. 179, 1791 |

| Sample | Compound | Sample Amount | Spk | Result | %R Calc | %R Reported | RPD Calc | RPD Reported | |
|---|---|---|---|---|---|---|---|---|---|
| MS (-012) | PFOS | 1280.3 | 25.60 | 1142.756 | -537.29 | -537 | 12.82 | 13 | p. 179 |
| MSD (-012) | PFOS | 1280.3 | 26.50 | 1299.243 | 71.47 | 71 | -- | -- | |
| LCS KQ2014290-01 | PFOS | -- | 29.70 | 37.10 | 124.92 | 125 | 0.81 | <1 | p. 185 |
| LCSD KQ2014290-02 | PFOS | -- | 29.70 | 37.40 | 125.93 | 126 | -- | -- | |
| LCS KQ2014290-01 | HFPO-DA | -- | 32.00 | 33.00 | 103.13 | 103 | 10.86 | 11 | p. 185 |
| LCSD KQ2014290-02 | HFPO-DA | -- | 32.00 | 29.60 | 92.50 | 93 | -- | -- | |

| | | Raw Data (ng/ml) | Calculated | Reported (ng/L) | Initial volume (L) | Final Volume (L) | | |
|---|---|---|---|---|---|---|---|---|
| Method Blank (KQ2014249-04): | PFOS | 0.004 | 0.14 | <0.44 | 0.25 | 0.008 | verified <0.44 | p. 137, 1172 |
| Instrument Blank (10/1/20 18:11): | PFOS | 0.0035 | 0.11 | U | 0.25 | 0.008 | verified <0.44 | p. 1845 |

AECOM
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Milwaukee, WI 53212
414.944.6080 tel
414.944.6081 fax

# Data Validation Report

| | |
|---|---|
| Project: | MCPA – Project 1007 |
| Laboratory: | ALS – Kelso, Washington |
| Work Order (WO): | K2008578 |
| Analyses/Method: | Polyfluorinated Alkyl Substances (PFASs)/PFAS by HPLC/MS/MS |
| Validation Level: | Stage 4 for 100% of samples |
| Prepared by: | Lisa Smith (CEAC) Completed on: 11/20/2020 |

The samples listed below were collected by AECOM at the Project 1007 Site on September 16 through 20, 2020.

| Sample ID | Quality Control | Sample Date | Laboratory ID |
|---|---|---|---|
| RC3-WAT-BULK-01-091620-U | | 9/16/2020 | K2008578-001 |
| RC3-WAT-BULK-02-091620-U | Field Duplicate | 9/16/2020 | K2008578-002 |
| RC3A-WAT-BULK-01-091920-U | | 9/19/2020 | K2008578-003 |
| RC3A-WAT-BULK-01-091920-F | | 9/19/2020 | K2008578-004 |
| RC3A-POWAT-01-092020-F | | 9/20/2020 | K2008578-005 |
| RC4A-WAT-BULK-01-091820-U | | 9/18/2020 | K2008578-606 |
| RC4A-WAT-BULK-01-091820-F | | 9/18/2020 | K2008578-007 |
| RC4A-POWAT-01-091820-F | | 9/18/2020 | K2008578-008 |
| RC5A-BULK-WAT-01-091820-U | | 9/18/2020 | K2008578-009 |
| RC5A-BULK-WAT-01-091820-F | | 9/18/2020 | K2008578-010 |
| RC5A-POWAT-01-0-6-01-091820 | | 9/16/2020 | K2008578-011 |
| RC3-WAT-BULK-03-091620 | Field Triplicate; MS/MSD | 9/16/2020 | K2008578-012 |
| **Field QC Samples:** | | | |
| TRIP BLANK 1 | Trip Blank | 9/16/2020 | K2008578-013 |
| TRIP BLANK 2 | Trip Blank | 9/16/2020 | K2008578-014 |

Data validation activities were conducted with reference to:

- Guidance for Perfluorochemicals Analysis, Minnesota Pollution Control Agency (MPCA), July 2020.
- Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020.

Control limits noted for this review were those from the MPCA Guidance for Perfluorochemicals Analysis, July 2020 and were used to evaluate whether quality control criteria were met.

**REVIEW ELEMENTS**

The data were evaluated based on the following parameters (where applicable to the method):

| | |
|---|---|
| ✓ | Data completeness (chain-of-custody (COC)/sample integrity |
| ✓ | Sample Receipt and Holding times |
| ✗ | Initial calibration/continuing calibration verification |
| ✗ | Laboratory and field blanks |
| ✗ | Labeled Analogs (Surrogates) |
| ✓ | Internal Standards |
| ✗ | Matrix spike (MS) and/or matrix spike duplicate (MSD) results |
| ✗ | Laboratory control sample (LCS) |
| ✗ | Field duplicates |
| ✓ | Quantitation and Result Verifications |

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

**SUMMARY**

Based on the results of the validation, the data are valid as reported and may be used for decision making purposes. Data qualifications were required as listed in Table 1. Results reported below the reporting limit (RL) were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the validator, but are not shown in Table 1.

**DETAILED REVIEW**

**Data Completeness**

The analytical data package was verified as complete, with the exception that raw instrument printouts were not provided for the initial calibrations. Summary forms were reviewed.

**Sample Receipt and Holding Times**

Samples were received at the laboratory intact and within the temperature requirement of <6 °C. Two trip blanks were received; however, were not listed on the chain of custody (COC).

Samples were extracted within the 14-day holding time, and analyzed within 28 days of extraction, with the exception of sample RC5A-POWAT-01-0-6-01-091820-F (K2008578-011). The sample was re-extracted 22 days over hold time. Associated detects (PFDS) were qualified as estimated biased low (J-) and nondetects (EtFOSA, EtFOSAA, MeFOSAA, PFTeDA, and PFTrDA) were qualified UJ.

**Calibration**

Initial calibrations (ICALs) for the target compounds were performed using linear calibrations. Relative standard deviations (RSDs) were within 20% and correlation coefficients were within the criteria of ≥ 0.99.

Initial and continuing calibration verifications (ICVs and CCVs) were performed at the correct frequency, ICVs were within ±30 % Difference (%D) and CCVs were within the criteria of 70-130% recovery, except as listed in the table below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | EtFOSAA results not reported from this ICV. |
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>RC3-WAT-BULK-01-091620 (K2008578-001)<br>RC3-WAT-BULK-02-091620 (K2008578-002)<br>RC3-WAT-BULK-03-091620 (K2008578-012) |
| CCV KC2000521 (10/1/2020) 697611 | 8:2 FTS | 138 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV KC2000531 (10/6/20) 698189 | PFBS | 139 %R | 70-130% | Associated detects were qualified as estimated biased high (J+):<br><br>RC3A-WAT-BULK-01-091920<br>RC4A-POWAT-01-091820<br>RC5A-BULK-WAT-01-091820 |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | |
| | PFTeDA | 170 %R | 70-130% | |
| | NMeFOSE | 267 %R | 70-130% | |
| | NEtFOSE | 292 %R | 70-130% | |
| | 6:2 FTS | 134 %R | 70-130% | |
| CCV KC2000572 (10/23/20) 700485 | PFTeDA | 142 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| | EtFOSA | 143 %R | 70-130% | |
| | NEtFOSE | 185 %R | 70-130% | |
| CCV KC2000572 (10/28/20) 701226 | NEtFOSE | 133 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV KC2000537 (10/7/20 5:53) 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect. No qualifiers. |

One ICV and CCV were recalculated for PFOS during the validation. Results reported are verified to be correct.

**Laboratory and Field Blanks**

Method blanks were analyzed to assess laboratory contamination. Target analytes were not detected in method blanks, with the exception of those listed in the table below. Results within 10 times the blank concentration were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Qualifiers |
|---|---|---|---|---|
| KQ2014249-04 | 9/29/2020 9:47 | 6:2 FTS | 4.3 J | Results qualified B: RC3-WAT-BULK-01-091620 (K2008578-001) RC3-WAT-BULK-02-091620 (K2008578-002) RC3-WAT-BULK-03-091620 (K2008578-012) |
| | | 8:2 FTS | 0.16 J | Associated results were nondetect. |
| KQ2014290-03 | 9/29/2020 15:17 | MeFOSA | 0.51 J | Results qualified B: C5A-POWAT-01-0-6-01-091820 (K2008578-011) |
| | | PFDoDA | 1.4 J | |
| | | PFTeDA | 2.6 J | No associated results. |
| | | PFTrDA | 3.6 J | |
| KQ2014429-03 | 10/1/2020 7:40 | MeFOSA | 0.75 J | Results qualified B: RC4A-WAT-BULK-01-091820 (K2008578-007) |
| | | PFTeDA | 3.7 J | Results qualified B: RC5A-BULK-WAT-01-091820 (K2008578-010) RC4A-POWAT-01-091820 (K2008578-008) RC3A-WAT-BULK-01-091920 (K2008578-004) |
| | | PFBS | 0.3 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | N-EtFOSE | 0.93 J | |
| | | N-MeFOSE | 1.1 J | |
| KQ2014435-03 | 10/1/2020 9:03 | N-EtFOSAA | 1.3 J | RC3A-WAT-BULK-01-091920 (K2008578-003) |
| | | PFHpA | 0.96 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | N-EtFOSE | 0.16 J | Associated results were nondetect. |
| | | PFTrDA | 1.4 J | |
| KQ2014516-03 | 10/2/2020 7:03 | PFBA | 0.53 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | PFHpA | 0.73 J | |
| | | MeFOSA | 0.71 J | Associated results were nondetect. |
| KQ2016108-03 | 10/22/2020 8:46 | N-EtFOSE | 0.18 J | Associated results were nondetect. |
| | | MeFOSA | 0.62 J | No associated results. |
| | | PFOA | 0.99 J | |

Continuing calibration blanks (CCBs) were analyzed after the CCVs. CCB detections along with data qualifiers are listed below.

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Qualifiers |
|---|---|---|---|---|
| KQ2014505-02 | 10/1/20 18:11 | EtFOSA | 0.60 J | Results qualified B: RC3-WAT-BULK-03-091620 (K2008578-012) |
| KQ2014704-02 | 10/2/20 14:32 | PFOA | 0.36 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | Associated results were nondetect. |
| | | EtFOSA | 0.40 J | Associated results were nondetect. |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | Associated results were greater than 10 times the blank value. No qualifiers. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Qualifiers |
|---|---|---|---|---|
| | | PFTeDA | 3.1 J | Associated detects previous qualified due to method blanks. |
| | | MeFOSA | 0.58 J | Associated results were nondetect. |
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | MeFOSA | 0.62 J | Associated results were nondetect. |
| | | EtFOSAA | 0.92 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | No associated results. |
| KQ2016704-02 | 10/28/20 16:44 | PFOA | 0.44 J | No associated results. |

Two trip blanks were associated with this sample group. The trip blanks were nondetect, with the exception of Trip Blank 2 which had a PFHpA detection of 0.96 J ng/L. PFHpA sample concentrations were greater than 10 times the blank concentration and were acceptable without qualification.

**Labeled Analogs**

Labeled analogs are spiked into all field samples, field QC samples, and method QC samples and are used during quantitation and to evaluate accuracy. Recoveries (%Rs) were within acceptable MPCA limits of 50% to 200%, with the exception of samples listed below. Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Results qualified are also listed below.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC3-WAT-BULK-01-091620 | K2008578-001 | D5-EtFOSAA | 45 | J+ |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | 13C2-PFTeDA | 38 | J+ |
| | | 13C3-PFBS | 45 | J+ |
| | | D9-EtFOSE | 36 | UJ |
| RC4A-WAT-BULK-01-091820 | K2008578-006 | 13C2-4:2 FTS | 241 | No qualifier |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | 13C3-HFPO-DA | 49 | UJ |
| RC4A-POWAT-01-091820 | K2008578-008 | 13C2-PFTeDA | 44 | J+ |
| | | 13C3-PFBS | 47 | J+ |
| | | D7-MeFOSE | 46 | UJ |
| | | D9-EtFOSE | 33 | UJ |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | 13C2-4:2 FTS | 205 | No qualifier |
| | | 13C3-PFBS | 47 | J+ |
| | | D9-EtFOSE | 37 | J+ |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | 13C2-PFDoDA | 37 | J+ |
| | | 13C2-PFTeDA | 38 | UJ |
| | | D3-MeFOSA | 49 | J+ |
| | | D3-MeFOSAA | 29 | UJ |
| | | D5-EtFOSAA | 22 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | D9-EtFOSE | 31 | UJ |

**Internal Standards**

The recoveries for the internal standard 13C7-PFUnDA were within acceptable MPCA limits of 50% to 150%.

**MS/MSD Results**

Additional sample volume was provided for an MS/MSD for sample RC3-WAT-BULK-03-091620-U. MS/MSD recoveries and RPDs were within the MPCA control limits, and were acceptable except as noted in the table below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **RC3-WAT-BULK-03-091620-U:** | | | |
| 6:2 FTS | **-160/-135** | 70-130 | The detects for this sample (and the associated field duplicates) were qualified as estimated biased low (J-):<br><br>RC3-WAT-BULK-01-091620-U<br>RC3-WAT-BULK-02-091620-U<br>RC3-WAT-BULK-03-091620-U |
| PFHxS | **36/59** | 70-130 | |
| PFHpS | **42/57** | 70-130 | |
| EtFOSAA | 110/**131** | 70-130 | The detects for this sample (and the associated field duplicates) were qualified as estimated biased high (J+):<br><br>RC3-WAT-BULK-01-091620-U<br>RC3-WAT-BULK-02-091620-U<br>RC3-WAT-BULK-03-091620-U |
| PFBA | **-45/32** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOA | **-141/-110** | 70-130 | |
| PFOS | **-537**/71 | 70-130 | |

**LCS Results**

LCSs are analyzed to monitor the accuracy of the analytical method independent of matrix effects. Laboratory control sample duplicates (LCSDs) results were provided to assess laboratory precision. The LCS/LCSD %Rs and RPDs were within the MPCA limits except as listed below.

| LCS/LCSD IDs (Batch) | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014249-03 (697611) | 8:2 FTS | **132/--** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDS | **61/--** | 70-130 | Not associated with results reported. |

| LCS/LCSD IDs (Batch) | Compound | LCS % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFNS | **68/--** | 70-130 | UJ/J-<br>RC3-WAT-BULK-01-091620<br>RC3-WAT-BULK-02-091620<br>RC3-WAT-BULK-03-091620 |
| KQ2014249-03<br>698697 | 6:2 FTS | **184/--** | 70-130 | J+<br>RC3-WAT-BULK-01-091620<br>RC3-WAT-BULK-02-091620<br>RC3-WAT-BULK-03-091620 |
| KQ2014290-01/<br>KQ2014290-02<br>(698190) | 11Cl-PF3OUdS | 73/**68** | 70-130 | The associated result was previously qualified due to an ICV exceedance. No further qualifiers. |
| KQ2014290-01/<br>KQ2014290-02<br>(699134) | MeFOSAA | 122/**132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoDA | **146/133** | 70-130 | J+:<br>RC5A-POWAT-01-0-6-01-091820 |
| | PFPeS | **143/**125 | 70-130 | |
| | PFUnDA | 126/**131** | 70-130 | |
| KQ2014290-01/<br>KQ2014290-02<br>(699503) | PFDS | **67/58** | 70-130 | Not associated with results reported. |
| | PFTrDA | **148/157** | 70-130 | |
| KQ2014429-01/<br>KQ2014429-02<br>(698189) | 4:2 FTS | **137/**120 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **138/**125 | 70-130 | J+:<br>RC4A-POWAT-01-091820<br>RC5A-BULK-WAT-01-091820 |
| | PFBA | **134/**130 | 70-130 | J+:<br>RC3A-WAT-BULK-01-091920<br>RC4A-WAT-BULK-01-091820<br>RC4A-POWAT-01-091820<br>RC5A-BULK-WAT-01-091820 |
| KQ2014429-01/<br>KQ2014429-02<br>(699503) | PFPeS | **166/153** | 70-130 | J+:<br>RC3A-WAT-BULK-01-091920<br>RC4A-POWAT-01-091820<br>RC5A-BULK-WAT-01-091820<br>RC4A-WAT-BULK-01-091820 |
| | PFTrDA | **139/**109 | 70-130 | J+:<br>RC3A-WAT-BULK-01-091920<br>RC4A-POWAT-01-091820<br>RC5A-BULK-WAT-01-091820 |
| KQ2016108-01/<br>KQ2016108-02<br>(700485) | EtFOSA | **154/145** | 70-130 | Associated results were nondetect. No qualifiers. |
| | NEtFOSE | **187/226** | 70-130 | |
| | NMeFOSE | **137/153** | 70-130 | |
| | PFTeDA | **134/**124 | 70-130 | |
| KQ2014516-01/<br>KQ2014516-02<br>(698467) | 10:2 FTS | 120/**178** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | 119/**145** | 70-130 | J+:<br>RC3A-POWAT-01-092020 |
| KQ2014524-01/<br>KQ2014524-01<br>(698467) | PFPeS | 130/**133** | 70-130 | J+:<br>RC4A-WAT-BULK-01-091820<br>RC5A-BULK-WAT-01-091820 |

**Field Duplicate Results**

Field duplicates are collected to assess the overall precision of field sampling and laboratory analysis. Samples RC3-WAT-BULK-02-091620-U and RC3-WAT-BULK-03-091620-U were collected as duplicates of RC3-WAT-BULK-01-091620-U. RPDs for the field duplicates were within the 30 percent limit for water sample, or were within ± 2 times the RL, with the exception of those indicated in bold in the table below. Results associated with field imprecision were qualified as estimated (J).

| Analyte | **Parent Sample Result (ng/L)** | **Field Duplicate Result (ng/L)** | **Triplicate Result (ng/L)** | **RPD (%)** |
|---|---|---|---|---|
| **RC3-WAT-BULK-01-091620-U/RC3-WAT-BULK-02-091620-U/RC3-WAT-BULK-03-091620-U:** | | | | |
| 6:2 FTS | 29 | 25 | 68 | **106** |
| EtFOSA | 4.1 U | 4.4 U | 0.38 J | -- |
| EtFOSAA | 29 | 20 | 20 | **39** |
| HFPO-DA | 0.31 J | 4.4 U | 0.31 J | -- |
| PFBA | 310 | 290 | 280 | 10 |
| PFBS | 14 | 13 | 14 | 7.3 |
| RLPFDA | 8.4 | 6.9 | 7.4 | 20 |
| PFDS | 0.48 J | 0.31 J | 4.4 U | ±RL |
| PFHpA | 46 | 38 | 42 | 19 |
| PFHpS | 29 | 21 | 25 | **32** |
| PFHxA | 68 | 64 | 61 | 11 |
| PFHxS | 47 | 38 | 41 | 21 |
| PFNA | 6.7 | 5.2 | 7.1 | 30 |
| PFNS | 1.3 J | 4.4 U | 4.4 U | -- |
| PFOA | 510 | 460 | 460 | 10 |
| PFOS | 1500 | 1300 | 1300 | 15 |
| PFOSA | 19 | 14 | 16 | **31** |
| PFPeA | 24 | 22 | 22 | 8.8 |
| PFPeS | 19 | 14 | 16 | 31 |

**Quantitation and Result Verifications**

Raw data, including quantitation reports, mass abundance reports (and ion profiles), sample prep logs, and instrument run logs were review and results were accurately identified and quantitated, with no transcription errors. The raw data were reviewed and recalculated for PFOS. No anomalies were observed during the re-calculation and verification of the results. Re-calculations are included in Attachment A.

Multiple sample dilutions were performed due to high analyte concentrations. These results were reviewed, and results quantitated within the instrument calibration range and reported from the lowest dilution were generally selected for use.

Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL).

Quantitations were performed to include branched isomer where applicable. In addition, signal to noise ratios were greater than 10:1 for ions used for quantification.

## Qualified Analytical Results

Data qualifications for reportable results are listed in Table 1. Results reported below the RL were qualified as estimated (J) by the laboratory; qualifications of these results were accepted by the Validator; however, are not listed in the table below.

**Table 1 - Data Validation Summary of Qualified Data**

| Sample ID | Lab ID | Analyte | CAS Number | Sample Conc (ng/L) | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|---|---|
| RC3-WAT-BULK-01-091620 | K2008578-001 | 11Cl-PF3OUdS | 763051-92-9 | 4.1 | UJ | c |
| RC3-WAT-BULK-01-091620 | K2008578-001 | 6:2 FTS | 27619-97-2 | 29 | BJ | fd,l,mb,ms |
| RC3-WAT-BULK-01-091620 | K2008578-001 | EtFOSAA | 2991-50-6 | 29 | J+ | fd,ms,s |
| RC3-WAT-BULK-01-091620 | K2008578-001 | PFHpS | 375-92-8 | 29 | J- | fd,ms |
| RC3-WAT-BULK-01-091620 | K2008578-001 | PFHxS | 355-46-4 | 47 | J- | ms |
| RC3-WAT-BULK-01-091620 | K2008578-001 | PFNS | 68259-12-1 | 1.3 | J- | l |
| RC3-WAT-BULK-01-091620 | K2008578-001 | PFOSA | 754-91-6 | 19 | J | fd |
| RC3-WAT-BULK-02-091620 | K2008578-002 | 11Cl-PF3OUdS | 763051-92-9 | 4.4 | UJ | c |
| RC3-WAT-BULK-02-091620 | K2008578-002 | 6:2 FTS | 27619-97-2 | 25 | BJ | fd,l,mb,ms |
| RC3-WAT-BULK-02-091620 | K2008578-002 | EtFOSAA | 2991-50-6 | 20 | J+ | fd,ms |
| RC3-WAT-BULK-02-091620 | K2008578-002 | PFHpS | 375-92-8 | 21 | J- | fd,ms |
| RC3-WAT-BULK-02-091620 | K2008578-002 | PFHxS | 355-46-4 | 38 | J- | ms |
| RC3-WAT-BULK-02-091620 | K2008578-002 | PFNS | 68259-12-1 | 0.59 | UJ | l |
| RC3-WAT-BULK-02-091620 | K2008578-002 | PFOSA | 754-91-6 | 14 | J | fd |
| RC3A-WAT-BULK-01-091920 | K2008578-003 | EtFOSAA | 2991-50-6 | 10 | B | mb |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | NEtFOSE | 1691-99-2 | 0.13 | UJ | s |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFBA | 375-22-4 | 250 | J+ | l |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFBS | 375-73-5 | 11 | J+ | s |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFOA | 335-67-1 | 160 | J+ | c |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFPeS | 2706-91-4 | 15 | J+ | l |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFTeDA | 376-06-7 | 4.1 | BJ+ | c,cb,mb,s |
| RC3A-WAT-BULK-01-091920 | K2008578-004 | PFTrDA | 72629-94-8 | 2.2 | J+ | c,l |
| RC3A-POWAT-01-092020 | K2008578-005 | PFPeS | 2706-91-4 | 27 | J+ | l |
| RC4A-WAT-BULK-01-091820 | K2008578-006 | PFPeS | 2706-91-4 | 41 | J+ | l |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | 11Cl-PF3OUdS | 763051-92-9 | 4.1 | UJ | c |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | HFPO-DA | 13252-13-6 | 0.29 | UJ | s |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | MeFOSA | 31506-32-8 | 0.77 | JB | mb |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | PFBA | 375-22-4 | 1100 | J+ | l |
| RC4A-WAT-BULK-01-091820 | K2008578-007 | PFPeS | 2706-91-4 | 46 | J+ | l |
| RC4A-POWAT-01-091820 | K2008578-008 | 6:2 FTS | 27619-97-2 | 0.66 | J+ | c,l |
| RC4A-POWAT-01-091820 | K2008578-008 | NEtFOSE | 1691-99-2 | 0.13 | UJ | s |
| RC4A-POWAT-01-091820 | K2008578-008 | NMeFOSE | 24448-09-7 | 0.3 | UJ | s |

| Sample ID | Lab ID | Analyte | CAS Number | Sample Conc (ng/L) | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|---|---|
| RC4A-POWAT-01-091820 | K2008578-008 | PFBA | 375-22-4 | 860 | J+ | l |
| RC4A-POWAT-01-091820 | K2008578-008 | PFBS | 375-73-5 | 25 | J+ | s |
| RC4A-POWAT-01-091820 | K2008578-008 | PFOA | 335-67-1 | 540 | J+ | c |
| RC4A-POWAT-01-091820 | K2008578-008 | PFPeS | 2706-91-4 | 44 | J+ | l |
| RC4A-POWAT-01-091820 | K2008578-008 | PFTeDA | 376-06-7 | 4 | BJ+ | c,cb,mb,s |
| RC4A-POWAT-01-091820 | K2008578-008 | PFTrDA | 72629-94-8 | 1.8 | J+ | c,l |
| RC5A-BULK-WAT-01-091820 | K2008578-009 | PFPeS | 2706-91-4 | 43 | J+ | l |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | 6:2 FTS | 27619-97-2 | 0.69 | J+ | c,l |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | NEtFOSE | 1691-99-2 | 0.3 | J+ | c,s |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFBA | 375-22-4 | 920 | J+ | l |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFBS | 375-73-5 | 28 | J+ | s |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFOA | 335-67-1 | 610 | J+ | c |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFPeS | 2706-91-4 | 46 | J+ | l |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFTeDA | 376-06-7 | 3.4 | BJ+ | c,cb,mb |
| RC5A-BULK-WAT-01-091820 | K2008578-010 | PFTrDA | 72629-94-8 | 1.3 | J+ | c,l |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | EtFOSAA | 2991-50-6 | 11 | J+ | s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | EtFOSA | 4151-50-2 | 0.27 | UJ | h |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | MeFOSA | 31506-32-8 | 0.59 | BJ+ | mb,s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | MeFOSAA | 2355-31-9 | 1.4 | UJ | s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | NEtFOSE | 1691-99-2 | 0.13 | UJ | h,s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | NMeFOSE | 24448-09-7 | 0.3 | UJ | h |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFDoA | 307-55-1 | 2.8 | BJ+ | l,mb,s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFDS | 335-77-3 | 0.51 | J- | h |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFPeS | 2706-91-4 | 23 | J+ | l |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFTeDA | 376-06-7 | 2 | UJ | h,s |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFTrDA | 72629-94-8 | 1.3 | UJ | h |
| RC5A-POWAT-01-0-6-01-091820 | K2008578-011 | PFUnA | 2058-94-8 | 1.8 | J+ | l |
| RC3-WAT-BULK-03-091620 | K2008578-012 | 11Cl-PF3OUdS | 763051-92-9 | 4.4 | UJ | c |
| RC3-WAT-BULK-03-091620 | K2008578-012 | 6:2 FTS | 27619-97-2 | 68 | BJ | fd,l,mb,ms |
| RC3-WAT-BULK-03-091620 | K2008578-012 | EtFOSA | 4151-50-2 | 0.38 | JB | cb |
| RC3-WAT-BULK-03-091620 | K2008578-012 | EtFOSAA | 2991-50-6 | 20 | J+ | fd,ms |
| RC3-WAT-BULK-03-091620 | K2008578-012 | PFHpS | 375-92-8 | 25 | J- | fd,ms |

| Sample ID | Lab ID | Analyte | CAS Number | Sample Conc (ng/L) | Validation Qualifier (1) | Reason Code (2) |
|---|---|---|---|---|---|---|
| RC3-WAT-BULK-03-091620 | K2008578-012 | PFHxS | 355-46-4 | 41 | J- | ms |
| RC3-WAT-BULK-03-091620 | K2008578-012 | PFNS | 68259-12-1 | 0.59 | UJ | l |
| RC3-WAT-BULK-03-091620 | K2008578-012 | PFOSA | 754-91-6 | 16 | J | fd |

(1): Data Validation Qualifiers:

| | |
|---|---|
| B | The analyte was detected in the sample at a concentration within ten times the blank concentration. |
| J | Estimated (+/- indicates high or low bias) |
| UJ | Estimated reporting limit |

(2): Reason Codes:

| | |
|---|---|
| c | Calibration |
| cb | Calibration blank |
| fd | Field duplicate |
| l | Laboratory control sample |
| mb | Method blank |
| ms | Matrix spike |
| s | Surrogate (labeled analog) |

**Attachment A**

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008579 Report date (mm/dd/yyyy): 10/28/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/25/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Three samples were re-extracted 22 days over holding time. See data qualification summary for qualifications. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank was nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Three results rejected due to severly low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP9A-WAT-MID-03-091920-U (K2008579-008) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD; MSD<br>FDs: EP9A-WAT-MID-01-091920/EP9A-WAT-MID-02-091920/EP9A-WAT-MID-03-091920 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>MSD - RPDs ok<br>LCSD and FD - See data qualification summary for summary of exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Three results rejected due to severly low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/25/2020

## Data Validation Qualifications for K2008579

**Holding Times (Section 1G):** Three samples were re-extracted 22 days over hold time. The results reported from the re-extractions were nondetect and qualified UJ. The samples and analytes qualified are listed below.

| Field Sample ID | Lab ID | Compounds |
|---|---|---|
| WL2-WAT-MID-01-091720 | K2008579-013 | EtFOSA |
| WL2-WAT-MID-01-091720 | K2008579-013 | NEtFOSE |
| WL2-WAT-MID-01-091720 | K2008579-013 | NMeFOSE |
| WL2-WAT-MID-01-091720 | K2008579-013 | PFDS |
| WL2-WAT-MID-01-091720 | K2008579-013 | PFTrDA |
| WL7-WAT-BULK-01-091720 | K2008579-015 | EtFOSA |
| WL7-WAT-BULK-01-091720 | K2008579-015 | NEtFOSE |
| WL7-WAT-BULK-01-091720 | K2008579-015 | NMeFOSE |
| WL7-WAT-BULK-01-091720 | K2008579-015 | PFDS |
| WL7-WAT-BULK-01-091720 | K2008579-015 | PFTrDA |
| WL5-WAT-MID-01-091720 | K2008579-017 | EtFOSA |
| WL5-WAT-MID-01-091720 | K2008579-017 | NEtFOSE |
| WL5-WAT-MID-01-091720 | K2008579-017 | NMeFOSE |
| WL5-WAT-MID-01-091720 | K2008579-017 | PFDS |
| WL5-WAT-MID-01-091720 | K2008579-017 | PFTrDA |

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>EP16-WAT-BULK-01-091620 (K2008579-011) |
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>EP16-WAT-BULK-01-091620 (K2008579-011) |
| CCV KC2000537 10/07/20 05:53 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000521 10/01/20 18:00 | 8:2 FTS | 138 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV K2008577 (10/06/20 19:30) 698188 | PFOA | 151 %R | 70-130% | J+:<br>WL7-WAT-BULK-01-091720 (K2008579-014)<br>WL5-WAT-MID-01-091720 (K2008579-016) |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | Results not reported from this CCV. |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | J+:<br>EP21A-WAT-MID-01-091920 (K2008579-010) |

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| | PFTeDA | 170 %R | 70-130% | J+:<br>EP14-WAT-MID-01-091920 (K2008579-004)<br>EP21A-WAT-MID-01-091920 (K2008579-010) |
| | NEtFOSE | 292 %R | 70-130% | J+:<br>EP14-WAT-MID-01-091920 (K2008579-004)<br>EP9A-WAT-MID-01-091920 (K2008579-006) |
| | NMeFOSE | 267 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | 6:2 FTS | 134 %R | 70-130% | |
| CCV KC2000572 (10/23/20 5:52) 700485 | PFTeDA | 142 %R | 70-130% | Results not reported from this CCV. |
| | EtFOSA | 143 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | NEtFOSE | 185 %R | 70-130% | |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014249-04 | 9/29/20 9:47 | 6:2 FTS | 4.3 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | 8:2 FTS | 0.16 J | Associated results were nondetect. |
| KQ2014290-03 | 9/29/2020 15:17 | MeFOSA | 0.51 J | WL2-WAT-MID-01-091720 (K2008579-013) |
| | | PFDoDA | 1.4 J | WL5-WAT-MID-01-091720 (K2008579-017)<br>WL7-WAT-BULK-01-091720 (K2008579-015) |
| | | PFTeDA | 2.6 J | WL2-WAT-MID-01-091720 (K2008579-013)<br>WL5-WAT-MID-01-091720 (K2008579-017)<br>WL7-WAT-BULK-01-091720 (K2008579-015) |
| | | PFTrDA | 3.6 J | Associated results were nondetect. |
| KQ2014429-03 | 10/1/2020 7:40 | MeFOSA | 0.75 J | EP14-WAT-MID-01-091920 (K2008579-004)<br>EP9A-WAT-MID-01-091920 (K2008579-006) |
| | | PFTeDA | 3.7 J | EP14-WAT-MID-01-091920 (K2008579-004)<br>EP21A-WAT-MID-01-091920 (K2008579-010) |
| | | PFBS | 0.3 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | NEtFOSE | 0.93 J | EP14-WAT-MID-01-091920 (K2008579-004)<br>EP9A-WAT-MID-01-091920 (K2008579-006) |
| | | NMeFOSE | 1.1 J | Associated results were nondetect or greater than 10 times the blank value. No qualifiers. |
| KQ2014435-03 | 10/1/20 9:03 | EtFOSAA | 1.3 J | EP14-WAT-MID-01-091920 (K2008579-003) |
| | | NEtFOSE | 0.16 J | EP9A-WAT-MID-01-091920 (K2008579-005) |
| | | PFHpA | 0.96 J | EP14-WAT-MID-01-091920 (K2008579-003)<br>EP9A-WAT-MID-01-091920 (K2008579-005)<br>EP9A-WAT-MID-02-091920 (K2008579-007)<br>EP9A-WAT-MID-03-091920 (K2008579-008)<br>EP21A-WAT-MID-01-091920 (K2008579-009) |
| | | PFTrDA | 1.4 J | Associated results were nondetect or greater than 10 times the blank value. No qualifiers. |
| KQ2014516-03 | 10/2/20 | MeFOSA | 0.71 J | Associated results were nondetect. |

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| | 7:03 | PFBA | 0.53 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| KQ2016108-03 | 10/22/2020 8:46 | N-EtFOSE | 0.18 J | Associated results were nondetect. |
| | | MeFOSA | 0.62 J | No associated results. |
| | | PFOA | 0.99 J | No associated results. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014505-02 | 10/1/20 18:11 | EtFOSA | 0.60 J | Associated results were nondetect. |
| KQ2014704-02 | 10/2/20 14:32 | PFOA | 0.36 J | No associated results. |
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | No associated results. |
| | | EtFOSA | 0.40 J | No associated results. |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | EP14-WAT-MID-01-091920 (K2008579-004) EP9A-WAT-MID-01-091920 (K2008579-006) EP21A-WAT-MID-01-091920 (K2008579-010) |
| | | PFTeDA | 3.1 J | EP14-WAT-MID-01-091920 (K2008579-004) EP21A-WAT-MID-01-091920 (K2008579-010) |
| | | MeFOSA | 0.58 J | EP14-WAT-MID-01-091920 (K2008579-004) EP9A-WAT-MID-01-091920 (K2008579-006) |
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | MeFOSA | 0.62 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | EtFOSAA | 0.92 J | EP14-WAT-MID-01-091920 (K2008579-003) |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | No associated results. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Three results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP9A-WAT-MID-01-091920 | K2008579-006 | D9-EtFOSE | 44 | J+ |
| EP21A-WAT-MID-01-091920 | K2008579-010 | 13C2-PFTeDA | 46 | J+ |
| | | D9-EtFOSE | 38 | UJ |
| WL2-WAT-MID-01-091720 | K2008579-012 | 13C2-4:2 FTS | 282 | No qualifier |
| WL2-WAT-MID-01-091720 | K2008579-013 | 13C2-PFTeDA | 4 | R |
| | | D3-MeFOSA | 41 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | D3-MeFOSAA | 26 | UJ |
| | | D5-EtFOSAA | 25 | UJ |
| | | D9-EtFOSE | 32 | J+ |
| | | 13C2-PFDoDA | 35 | UJ |
| | | 13C2-PFUnDA | 47 | UJ |
| WL7-WAT-BULK-01-091720 | K2008579-014 | 13C2-4:2 FTS | 321 | No qualifier |
| | | 13C2-6:2 FTS | 227 | No qualifier |
| WL7-WAT-BULK-01-091720 | K2008579-015 | 13C2-PFTeDA | 7 | R |
| | | D3-MeFOSA | 43 | UJ |
| | | D3-MeFOSAA | 31 | UJ |
| | | D5-EtFOSAA | 30 | UJ |
| | | D9-EtFOSE | 42 | UJ |
| | | 13C2-PFDoDA | 40 | J+ |
| WL5-WAT-MID-01-091720 | K2008579-016 | 13C2-4:2 FTS | 303 | No qualifier |
| | | 13C2-6:2 FTS | 219 | No qualifier |
| WL5-WAT-MID-01-091720 | K2008579-017 | 13C2-PFTeDA | 5 | R |
| | | D3-MeFOSA | 44 | UJ |
| | | D3-MeFOSAA | 30 | UJ |
| | | D5-EtFOSA | 28 | UJ |
| | | D5-EtFOSAA | 24 | UJ |
| | | D9-EtFOSE | 34 | UJ |
| | | 13C2-PFDoDA | 32 | J+ |
| | | 13C2-PFUnDA | 47 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) and detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014290-01/ KQ2014290-02 (698190) | 11Cl-PF3OUdS | 73/**68** | 70-130 | Associated results were nondetect and qualified UJ:<br><br>WL2-WAT-MID-01-091720-F (K2008579-013 )<br>WL7-WAT-BULK-01-091720-F (K2008579-015)<br>WL5-WAT-MID-01-091720-F (K2008579-017) |
| KQ2014290-01/ KQ2014290-02 (699134) | MeFOSAA | 122/**132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoDA | **146/133** | 70-130 | J+:<br>WL7-WAT-BULK-01-091720 (K2008579-015)<br>WL5-WAT-MID-01-091720 (K2008579-017) |
| | PFUnDA | 126/**131** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | **143/**125 | 70-130 | J+:<br>WL2-WAT-MID-01-091720 (K2008579-013)<br>WL7-WAT-BULK-01-091720 (K2008579-015)<br>WL5-WAT-MID-01-091720 (K2008579-017) |
| KQ2014290-01/ KQ2014290-02 (699503) | PFDS | **67/58** | 70-130 | PFDS sample results not reported from this LCS. |
| | PFTrDA | **148/157** | 70-130 | Associated results were nondetect. No qualifiers. |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014429-01/ KQ2014429-02 (698189) | 4:2 FTS | **137/**120 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **138/**125 | 70-130 | |
| | PFBA | **134/**130 | 70-130 | J+:<br>EP14-WAT-MID-01-091920 (K2008579-004)<br>EP9A-WAT-MID-01-091920 (K2008579-006)<br>EP21A-WAT-MID-01-091920 (K2008579-010) |
| KQ2014429-01/ KQ2014429-02 (699503) | PFPeS | **166/153** | 70-130 | J+:<br>EP14-WAT-MID-01-091920 (K2008579-004)<br>EP9A-WAT-MID-01-091920 (K2008579-006)<br>EP21A-WAT-MID-01-091920 (K2008579-010) |
| | PFTrDA | **139/**109 | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014249-03 | 6:2 FTS | **184/**-- | 70-130 | J+:<br>EP16-WAT-BULK-01-091620 (K2008579-011) |
| | 8:2 FTS | **132/**-- | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFNS | **68/**-- | 70-130 | Associated results were nondetect and qualified UJ:<br>EP16-WAT-BULK-01-091620 (K2008579-011) |
| | PFDS | **61/**-- | 70-130 | |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 120/**178** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | 119/**145** | 70-130 | J+:<br>EP20-WAT-BULK-01-092020-F (K2008579-002) |
| KQ2016108-01/ KQ2016108-02 (700485) | EtFOSA | **154/145** | 70-130 | Associated results were nondetect. No qualifiers. |
| | NEtFOSE | **187/226** | 70-130 | |
| | NMeFOSE | **137/153** | 70-130 | |
| | PFTeDA | **134/**124 | 70-130 | PFTeDA sample results not reported from this LCS. |
| KQ2014524-01/ KQ2014524-01 (698467) | PFPeS | 130/**133** | 70-130 | J+:<br>EP20-WAT-BULK-01-092020 (K2008579-001) |

**Matrix spike/Matrix spike duplicate (Section 6Av):** Results associated with low recoveries were qualified as estimated biased low (J-) and detects associated with high recoveries were qualified as J+:

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **EP9A-WAT-MID-03-091920-U:** | | | |
| PFBA | 113/**38** | 70-130 | The detects for this sample (and the associated field duplicates) were qualified as estimated biased low (J-):<br><br>EP9A-WAT-MID-01-091920 (K2008579-006)<br>EP9A-WAT-MID-02-091920 (K2008579-007)<br>EP9A-WAT-MID-03-091920 (K2008579-008) |
| PFOS | **137/**97 | 70-130 | The detects for this sample (and the associated field duplicates) were qualified as estimated biased high (J+):<br><br>EP9A-WAT-MID-01-091920 (K2008579-006)<br>EP9A-WAT-MID-02-091920 (K2008579-007)<br>EP9A-WAT-MID-03-091920 (K2008579-008) |
| PFPeS | **175/156** | 70-130 | |
| PFTrDA | **164/150** | 70-130 | Associated results were nondetect and were acceptable without qualification. |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 39 | 30 | Associated results were nondetect. No qualifiers. |
| | PFHpS | 35 | 30 | J+: EP20-WAT-BULK-01-092020 (K2008579-002) |

**Field duplicate RPDs (Section 6Bi):** PFOS detects for samples EP9A-WAT-MID-01-091920 (K2008579-006), EP9A-WAT-MID-02-091920 (K2008579-007), and EP9A-WAT-MID-03-091920 (K2008579-008) were qualified as estimated (J) due to an RPD of 36%.

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008580 Report date (mm/dd/yyyy): 10/27/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/27/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Trip blank noted as being extracted over hold time. No qualifiers as the trip blank sample date is usually set as that of the earliest sample collected. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank was nondetect. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Twenty-one results rejected due to severly low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for summary of exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Twenty-one results rejected due to severly low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/27/2020

## Data Validation Qualifications for K2008580

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>EP27C-WAT-BOT-01-091520 (K2008580-005)<br>EP27C-WAT-MID-01-091520 (K2008580-007)<br>EP18-WAT-MID-01-091520 (K2008580-009)<br>EP8-WAT-BULK-01-091620 (K2008580-013) |
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were qualified as estimated (UJ/J):<br>EP27C-WAT-BOT-01-091520 (K2008580-005)<br>EP27C-WAT-MID-01-091520 (K2008580-007)<br>EP18-WAT-MID-01-091520 (K2008580-009)<br>EP8-WAT-BULK-01-091620 (K2008580-013) |
| CCV KC2000537 10/07/20 05:53 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000521 10/01/20 18:00 | 8:2 FTS | 138 %R | 70-130% | J+:<br>EP27C-WAT-MID-01-091520 (K2008580-007) |
| CCV K2008577 (10/06/20 19:30) 698188 | PFOA | 151 %R | 70-130% | J+:<br>EP26A-WAT-BULK-01-091720 (K2008580-001) |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | Results not reported from this CCV. |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | J+:<br>EP19-WAT-BULK-01-091920 (K2008580-004) |
| | PFTeDA | 170 %R | 70-130% | |
| | NEtFOSE | 292 %R | 70-130% | |
| | NMeFOSE | 267 %R | 70-130% | J+:<br>EP26A-WAT-BULK-01-091720 (K2008580-002)<br>EP20A-POWAT-01-091720 (K2008580-014) |
| | 6:2 FTS | 134 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000572 10/23/20 00:43 700484 | PFTeDA | 141 %R | 70-130% | Results not reported from this CCV. |
| | EtFOSAA | 59.8 %R | 70-130% | |
| CCV KC2000572 10/23/20 03:23 700484 | PFTeDA | 142 %R | 70-130% | Results not reported from this CCV. |
| CCV KC2000572 (10/23/20 5:52) 700484 | PFTeDA | 142 %R | 70-130% | Results not reported from this CCV. |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014249-04 | 9/29/20 9:47 | 6:2 FTS | 4.3 J | EP18-WAT-MID-01-091520 (K2008580-009) EP8-WAT-BULK-01-091620 (K2008580-013) |
| | | 8:2 FTS | 0.16 J | EP27C-WAT-MID-01-091520 (K2008580-007) |
| KQ2014275-03 | 9/29/2020 12:47 | PFHpA | 0.98 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | MeFOSA | 0.79 J | No associated results. |
| KQ2014429-03 | 10/1/2020 7:40 | MeFOSA | 0.75 J | EP26A-WAT-BULK-01-091720 (K2008580-002) |
| | | PFTeDA | 3.7 J | EP19-WAT-BULK-01-091920 (K2008580-004) |
| | | NEtFOSE | 0.93 J | |
| | | PFBS | 0.3 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | NMeFOSE | 1.1 J | EP26A-WAT-BULK-01-091720 (K2008580-002) EP20A-POWAT-01-091720 (K2008580-014) |
| KQ2014435-03 | 10/1/20 9:03 | EtFOSAA | 1.3 J | EP19-WAT-BULK-01-091920 (K2008580-003) |
| | | NEtFOSE | 0.16 J | Associated results were nondetect. |
| | | PFHpA | 0.96 J | Associated results were nondetect or greater than 10 times the blank value. No qualifiers. |
| | | PFTrDA | 1.4 J | EP19-WAT-BULK-01-091920 (K2008580-003) EP15-WAT-MID-01-091920 (K2008580-011) |
| KQ2014516-03 | 10/2/20 7:03 | MeFOSA | 0.71 J | Associated results were nondetect. |
| | | PFBA | 0.53 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | PFHpA | 0.73 J | |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014505-02 | 10/1/20 18:11 | EtFOSA | 0.60 J | Associated results were nondetect. |
| KQ2014704-02 | 10/2/20 14:32 | PFOA | 0.36 J | No associated results. |
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | EP26A-WAT-BULK-01-091720 (K2008580-001) EP27C-WAT-BOT-01-091520 (K2008580-006) EP27C-WAT-MID-01-091520 (K2008580-008) EP18-WAT-MID-01-091520 (K2008580-010) |
| | | EtFOSA | 0.40 J | Associated results were nondetect. |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | PFTeDA | 3.1 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | MeFOSA | 0.58 J | Associated results were nondetect or greater than 10 times the blank value. No qualifiers. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | Associated results were nondetect or greater than 10 times the blank value. No qualifiers. |
| | | MeFOSA | 0.62 J | Associated results were nondetect. |
| | | EtFOSAA | 0.92 J | EP19-WAT-BULK-01-091920 (K2008580-003) EP19-POWAT-01-092020 (K2008580-015) |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |
| KQ2016364-02 | 10/23/20 00:53 | PFOA | 0.94 J | No associated results. |
| KQ2016364-08 | 10/23/20 03:33 | PFOA | 0.55 J | No associated results. |
| | | EtFOSAA | 1.1 J | |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Twenty-one results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP26A-WAT-BULK-01-091720 | K2008580-001 | 13C2-4:2 FTS | 247 | No Qualifier |
| | | 13C2-PFDoDA | 43 | J+ |
| | | 13C2-PFTeDA | 17 | R |
| | | D3-MeFOSAA | 33 | UJ |
| | | D5-EfFOSAA | 29 | J+ |
| | | D9-EtFOSE | 37 | J+ |
| EP26A-WAT-BULK-01-091720 | K2008580-002 | 13C2-8:2 FTS | 34 | UJ |
| | | 13C2-PFDA | 20 | J+ |
| | | 13C2-PFDoDA | 7 | R |
| | | 13C2-PFTeDA | 2 | R |
| | | 13C2-PFUnDA | 11 | R |
| | | 13C4-PFOS | 27 | J+ |
| | | 13C8-FOSA | 12 | R |
| | | D3-MeFOSA | 4 | R |
| | | D3-MeFOSAA | 10 | R |
| | | D5-EfFOSAA | 5 | R |
| | | D5-EtFOSA | 2 | R |
| | | D7-MeFOSE | 2 | R |
| | | D9-EtFOSE | 1 | R |
| EP19-WAT-BULK-01-091920 | K2008580-003 | 13C2-4:2 FTS | 247 | No Qualifier |
| EP19-WAT-BULK-01-091920 | K2008580-004 | 13C2-4:2 FTS | 212 | No Qualifier |
| | | D9-EtFOSE | 42 | J+ |
| EP27C-WAT-BOT-01-091520 | K2008580-006 | 13C2-4:2 FTS | 247 | No Qualifier |
| | | D9-EtFOSE | 48 | UJ |
| EP27C-WAT-MID-01-091520 | K2008580-008 | 13C2-4:2 FTS | 205 | No Qualifier |
| | | 13C2-PFTeDA | 38 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | D9-EtFOSE | 35 | J+ |
| EP18-WAT-MID-01-091520 | K2008580-009 | 13C2-PFTeDA | 47 | UJ |
| EP18-WAT-MID-01-091520 | K2008580-010 | 13C2-4:2 FTS | 245 | No Qualifier |
| | | 13C2-PFTeDA | 42 | J+ |
| | | D9-EtFOSE | 37 | J+ |
| EP20A-POWAT-01-091720 | K2008580-014 | 13C2-8:2 FTS | 40 | J+ |
| | | 13C2-PFDA | 26 | J+ |
| | | 13C2-PFDoDA | 6 | R |
| | | 13C2-PFTeDA | 4 | R |
| | | 13C2-PFUnDA | 11 | R |
| | | 13C4-PFOS | 43 | J+ |
| | | 13C8-FOSA | 16 | R |
| | | D3-MeFOSA | 5 | R |
| | | D3-MeFOSAA | 8 | R |
| | | D5-EfFOSAA | 5 | R |
| | | D5-EtFOSA | 4 | R |
| | | D7-MeFOSE | 4 | R |
| | | D9-EtFOSE | 4 | R |
| EP19-POWAT-01-092020 | K2008580-015 | 13C2-PFTeDA | 48 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) and detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014429-01/ KQ2014429-02 (698189) | 4:2 FTS | **137/**120 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **138/**125 | 70-130 | |
| | PFBA | **134/**130 | 70-130 | J+:<br>EP26A-WAT-BULK-01-091720 (K2008580-002)<br>EP19-WAT-BULK-01-091920 (K2008580-004)<br>EP20A-POWAT-01-091720 (K2008580-014) |
| KQ2014429-01/ KQ2014429-02 (699503) | PFPeS | **166/153** | 70-130 | J+:<br>EP26A-WAT-BULK-01-091720 (K2008580-002)<br>EP19-WAT-BULK-01-091920 (K2008580-004)<br>EP20A-POWAT-01-091720 (K2008580-014) |
| | PFTrDA | **139/**109 | 70-130 | J+:<br>EP26A-WAT-BULK-01-091720 (K2008580-002)<br>EP20A-POWAT-01-091720 (K2008580-014) |
| KQ2014249-03 | 6:2 FTS | **184/**-- | 70-130 | J+:<br>EP27C-WAT-BOT-01-091520 (K2008580-005)<br>EP27C-WAT-MID-01-091520 (K2008580-007)<br>EP18-WAT-MID-01-091520 (K2008580-009)<br>EP8-WAT-BULK-01-091620 (K2008580-013) |
| | 8:2 FTS | **132/**-- | 70-130 | J+:<br>EP27C-WAT-MID-01-091520 (K2008580-007) |
| | PFNS | **68/**-- | 70-130 | Associated results were nondetect and qualified UJ: |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFDS | **61/**-- | 70-130 | EP27C-WAT-BOT-01-091520 (K2008580-005)<br>EP27C-WAT-MID-01-091520 (K2008580-007)<br>EP18-WAT-MID-01-091520 (K2008580-009)<br>EP8-WAT-BULK-01-091620 (K2008580-013) |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 120/**178** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | 119/**145** | 70-130 | J+:<br>EP15-WAT-MID-01-091920 (K2008580-012)<br>EP19-POWAT-01-092020 (K2008580-015) |
| KQ2014275-01/ KQ2014275-02 (697977) | 10:2 FTS | 100/**140** | 70-130 | Associated results were nondetect. No qualifiers. |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 5Bii):** Detects associated with high RPDs were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014275-01/ KQ2014275-02 (697977) | 10:2 FTS | 33 | 30 | Associated results were nondetect. No qualifiers. |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 39 | 30 | Associated results were nondetect. No qualifiers. |
| | PFHpS | 35 | 30 | J+:<br>EP15-WAT-MID-01-091920 (K2008580-012)<br>EP19-POWAT-01-092020 (K2008580-015) |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008581 Report date (mm/dd/yyyy): 10/29/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/27/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Two samples were re-extracted 22 days over holding time. See data qualification summary for qualifications. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank detects were less than the RL:<br>PFOA 0.46 J ng/L<br>EtFOSA 0.3 J ng/L<br>6:2 FTS 2.3 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for discussion. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Two results rejected due to severly low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD<br>FDs: RC17A-WAT-BULK-01-091920/RC17A-WAT-BULK-02-091920 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | | | summary of exceedances and qualifications.<br>FD RPDs ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Two results rejected due to severly low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/27/2020

## Data Validation Qualifications for K2008581

**Holding Times (Section 1G):** Two samples were re-extracted 22 days over hold time. The results reported from the re-extractions were qualified as estimated (UJ/J-). The samples and analytes qualified are listed below.

| Field Sample ID | Lab ID | Compounds |
|---|---|---|
| RC18-WAT-BULK-01-091720 | K2008581-005 | EtFOSA |
| RC18-WAT-BULK-01-091720 | K2008581-005 | NEtFOSE |
| RC18-WAT-BULK-01-091720 | K2008581-005 | NMeFOSE |
| RC18-WAT-BULK-01-091720 | K2008581-005 | PFDS |
| RC18-WAT-BULK-01-091720 | K2008581-005 | PFTrDA |
| RC18-POWAT-01-091720 | K2008581-006 | EtFOSA |
| RC18-POWAT-01-091720 | K2008581-006 | NEtFOSE |
| RC18-POWAT-01-091720 | K2008581-006 | NMeFOSE |
| RC18-POWAT-01-091720 | K2008581-006 | PFDS |
| RC18-POWAT-01-091720 | K2008581-006 | PFTrDA |

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>EP1A-WAT-MID-01-091520 (K2008581-014)<br>EP1A-WAT-BOT-01-091520 (K2008581-012)<br>TRIP BLANK (K2008581-016) |
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were qualified as estimated (UJ/J):<br>EP1A-WAT-BOT-01-091520 (K2008581-012)<br>EP1A-WAT-MID-01-091520 (K2008581-014)<br>TRIP BLANK (K2008581-016) |
| CCV KC2000537 10/07/20 05:53 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000521 10/01/20 18:00 | 8:2 FTS | 138 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | Results not reported from this CCV. |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | |
| | PFTeDA | 170 %R | 70-130% | |
| | NEtFOSE | 292 %R | 70-130% | |
| | NMeFOSE | 267 %R | 70-130% | |
| | 6:2 FTS | 134 %R | 70-130% | |
| CCV KC2000572 (10/23/20 5:52) 700484 | PFTeDA | 142 %R | 70-130% | Results not reported from this CCV. |
| | EtFOSAA | 185 %R | 70-130% | |

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| | EtFOSA | 143 %R | 70-130% | Associated results were nondetect. No qualifiers. |

**Trip blank detections (Section 3Aii):** The 6:2 FTS and EtFOSA detections were determined to be laboratory contamination per qualifications in the next section. PFOA detects for the samples were greater than 10 times the trip blank concentration and were acceptable without qualification.

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014249-04 | 9/29/20 9:47 | 6:2 FTS | 4.3 J | EP1A-WAT-BOT-01-091520 (K2008581-012)<br>EP1A-WAT-MID-01-091520 (K2008581-014)<br>TRIP BLANK (K2008581-016) |
| | | 8:2 FTS | 0.16 J | Associated results were nondetect. |
| KQ2014275-03 | 9/29/2020 12:47 | PFHpA | 0.98 J | EP1A-WAT-BOT-01-091520 (K2008581-013) |
| | | MeFOSA | 0.79 J | EP1A-WAT-MID-01-091520 (K2008581-015) |
| KQ2014290-03 | 9/29/2020 15:17 | PFDoA | 1.4 J | RC18-WAT-BULK-01-091720 (K2008581-005)<br>RC18-POWAT-01-091720 (K2008581-006) |
| | | MeFOSA | 0.51 J | RC18-POWAT-01-091720 (K2008581-006) |
| | | PFTeDA | 2.6 J | RC18-WAT-BULK-01-091720 (K2008581-005)<br>RC18-POWAT-01-091720 (K2008581-006) |
| | | PFTrDA | 3.6 J | No associated results. |
| KQ2014435-03 | 10/1/2020 9:03 | EtFOSAA | 1.3 J | EP17A-WAT-BULK-01-091920 (K2008581-007) |
| | | NEtFOSE | 0.16 J | RC17-WAT-BULK-02-091920 (K2008581-011) |
| | | PFHpA | 0.96 J | RC16A-WAT-BULK-01-091920 (K2008581-001)<br>EP17A-WAT-BULK-01-091920 (K2008581-007)<br>RC17A-WAT-BULK-01-091920 (K2008581-009)<br>RC17-WAT-BULK-02-091920 (K2008581-011) |
| | | PFTrDA | 1.4 J | RC17A-WAT-BULK-01-091920 (K2008581-009) |
| KQ2014516-03 | 10/2/2020 7:03 | MeFOSA | 0.71 J | EP17A-WAT-BULK-01-091920 (K2008581-008) |
| | | PFBA | 0.53 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| | | PFHpA | 0.73 J | RC16A-WAT-BULK-01-091920 (K2008581-002)<br>EP17A-WAT-BULK-01-091920 (K2008581-008)<br>RC17A-WAT-BULK-01-091920 (K2008581-010) |
| KQ2016108-03 | 10/22/2020 8:46 | MeFOSA | 0.62 J | No associated results. |
| | | NEtFOSE | 0.18 J | RC18-POWAT-01-091720 (K2008581-006) |
| | | PFOA | 0.99 J | No associated results. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014505-02 | 10/1/20 18:11 | EtFOSA | 0.60 J | TRIP BLANK (K2008581-016) |
| KQ2014704-02 | 10/2/20 14:32 | PFOA | 0.36 J | No associated results. |
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | EP1A-WAT-MID-01-091520 (K2008581-015) |
| | | EtFOSA | 0.40 J | Associated results were nondetect. |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | No associated results. |
| | | PFTeDA | 3.1 J | |
| | | MeFOSA | 0.58 J | |
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | RC16A-WAT-BULK-01-091920 (K2008581-001)<br>EP17A-WAT-BULK-01-091920 (K2008581-007)<br>RC17A-WAT-BULK-01-091920 (K2008581-009)<br>RC17-WAT-BULK-02-091920 (K2008581-011)<br>RC16A-WAT-BULK-01-091920 (K2008581-002)<br>EP17A-WAT-BULK-01-091920 (K2008581-008)<br>RC17A-WAT-BULK-01-091920 (K2008581-010) |
| | | MeFOSA | 0.62 J | EP17A-WAT-BULK-01-091920 (K2008581-008) |
| | | EtFOSAA | 0.92 J | EP17A-WAT-BULK-01-091920 (K2008581-007)<br>RC17A-WAT-BULK-01-091920 (K2008581-010) |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | Associated results were nondetect. |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | Associated results were greater than 10 times the blank value. No qualifiers. |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | No associated results. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Two results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC18-WAT-BULK-01-091720 | K2008581-004 | 13C2-4:2 FTS | 298 | No Qualifier |
| | | 13C2-6:2 FTS | 224 | No Qualifier |
| | | 13C2-PFTeDA | 38 | J+ |
| | | D7-MeFOSE | 46 | UJ |
| | | D9-EtFOSE | 35 | UJ |
| RC18-WAT-BULK-01-091720 | K2008581-005 | 13C2-PFDoDA | 36 | J+ |
| | | 13C2-PFTeDA | 5 | R |
| | | D3-MeFOSA | 43 | UJ |
| | | D3-MeFOSAA | 27 | UJ |
| | | D5-EtFOSA | 48 | UJ |
| | | D5-EtFOSAA | 20 | UJ |
| | | D9-EtFOSE | 40 | UJ |
| RC18-POWAT-01-091720 | K2008581-006 | 13C2-PFDoDA | 39 | J+ |
| | | 13C2-PFTeDA | 7 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| | | D3-MeFOSA | 44 | J+ |
| | | D3-MeFOSAA | 27 | UJ |
| | | D5-EtFOSAA | 21 | J+ |
| EP17A-WAT-BULK-01-091920 | K2008581-007 | 13C2-4:2 FTS | 234 | No Qualifier |
| RC17-WAT-BULK-02-091920 | K2008581-011 | 13C2-4:2 FTS | 228 | No Qualifier |
| EP1A-WAT-BOT-01-091520 | K2008581-013 | 13C2-PFTeDA | 49 | J+ |
| | | D9-EtFOSE | 43 | J+ |
| EP1A-WAT-MID-01-091520 | K2008581-014 | 13C2-PFTeDA | 45 | UJ |
| EP1A-WAT-MID-01-091520 | K2008581-015 | 13C2-PFTeDA | 38 | J+ |
| | | D9-EtFOSE | 35 | J+ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) and detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014290-01/ KQ2014290-02 (698190) | 11Cl-PF3OUdS | 73/**68** | 70-130 | Associated result results were nondetect and qualified UJ:<br>RC18-WAT-BULK-01-091720 (K2008581-005)<br>RC18-POWAT-01-091720 (K2008581-006) |
| KQ2014290-01/ KQ2014290-02 (699134) | MeFOSAA | 122**/132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoDA | **146/133** | 70-130 | J+:<br>RC18-WAT-BULK-01-091720 (K2008581-005)<br>RC18-POWAT-01-091720 (K2008581-006) |
| | PFUnDA | 126/**131** | 70-130 | J+:<br>RC18-WAT-BULK-01-091720 (K2008581-005) |
| | PFPeS | **143/**125 | 70-130 | J+:<br>RC18-POWAT-01-091720 (K2008581-006) |
| KQ2014290-01/ KQ2014290-02 (699503) | PFDS | **67/58** | 70-130 | No associated results. |
| | PFTrDA | **148/157** | 70-130 | |
| KQ2014249-03 | 6:2 FTS | **184/**-- | 70-130 | J+:<br>EP1A-WAT-BOT-01-091520 (K2008581-012)<br>EP1A-WAT-MID-01-091520 (K2008581-014)<br>TRIP BLANK (K2008581-016) |
| | 8:2 FTS | **132/**-- | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFNS | **68/**-- | 70-130 | Associated result results were nondetect and qualified UJ:<br>EP1A-WAT-BOT-01-091520 (K2008581-012)<br>EP1A-WAT-MID-01-091520 (K2008581-014)<br>TRIP BLANK (K2008581-016) |
| | PFDS | **61/**-- | 70-130 | |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 120/**178** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | 119/**145** | 70-130 | J+:<br>EP17A-WAT-BULK-01-091920 (K2008581-008) |
| KQ2014275-01/ KQ2014275-02 (697977) | 10:2 FTS | 100/**140** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014635-01/ | PFDoDA | 129/**149** | 70-130 | Associated results were nondetect. No qualifiers. |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014635-02 (698705) | PFHpS | **132/136** | 70-130 | |
| | PFPeS | **146/141** | 70-130 | |
| | PFUnDA | 125**/134** | 70-130 | |
| | PFOS | 127**/132** | 70-130 | J+: RC16A-POWAT-01-092120 (K2008581-003) |
| KQ2014635-01/ KQ2014635-02 (698706) | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2016108-01/ KQ2016108-02 (700485) | EtFOSA | **154/145** | 70-130 | Associated results were nondetect. No qualifiers. |
| | NMeFOSE | **137/153** | 70-130 | |
| | PFTeDA | **134/**124 | 70-130 | |
| | NEtFOSE | **187/226** | 70-130 | J+: RC18-POWAT-01-091720 (K2008581-006) |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014275-01/ KQ2014275-02 (697977) | 10:2 FTS | 33 | 30 | Associated results were nondetect. No qualifiers. |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 39 | 30 | Associated results were nondetect. No qualifiers. |
| | PFHpS | 35 | 30 | J: EP17A-WAT-BULK-01-091920 (K2008581-008) |

Any R qualifiers take precedence over other qualifiers.

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008582 Report date (mm/dd/yyyy): 10/30/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/28/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but not was listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☒ | ☐ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☒ | ☐ | ☐ | Seven samples were re-extracted 22 days over holding time. See data qualification summary for qualifications. |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank detects were less than the RL:<br>PFHpA 0.78 J ng/L<br>PFTeDA 2.3 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for discussion. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Three results rejected due to severly low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☒ | ☐ | ☐ | 30% limit for PFAS (water).<br>LCSD - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Three results rejected due to severly low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/28/2020

## Data Validation Qualifications for K2008582

**Holding Times (Section 1G):** Seven samples samples were re-extracted 22 days over hold time. Nondetect results reported from the re-extractions were qualified UJ and detects were qualified as estimated biased low (J-). The samples and analytes qualified are listed below.

| Field Sample ID | Lab ID | Compounds | Qualifier |
|---|---|---|---|
| WL15-WAT-MID-01-091620 | K2008582-002 | PFTrDA | UJ |
| WL15-WAT-MID-01-091620 | K2008582-002 | PFTeDA | UJ |
| WL15-WAT-MID-01-091620 | K2008582-002 | EtFOSA | UJ |
| WL15-WAT-MID-01-091620 | K2008582-002 | NMeFOSE | UJ |
| WL15-WAT-MID-01-091620 | K2008582-002 | NEtFOSE | UJ |
| WL15-WAT-MID-01-091620 | K2008582-002 | PFDS | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | PFTrDA | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | NEtFOSE | J- |
| WL6-WAT-BULK-01-091720 | K2008582-004 | NMeFOSE | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | PFTeDA | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | PFDS | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | EtFOSA | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | NEtFOSE | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | EtFOSA | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | PFDS | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | PFTeDA | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | NMeFOSE | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | PFTrDA | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | PFTrDA | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | PFTeDA | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | EtFOSA | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | NMeFOSE | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | NEtFOSE | UJ |
| WL21-WAT-MID-01-091620 | K2008582-010 | PFDS | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | PFDS | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | NEtFOSE | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | NMeFOSE | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | EtFOSA | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | PFTeDA | UJ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | PFTrDA | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | PFTrDA | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | PFTeDA | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | EtFOSA | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | NMeFOSE | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | NEtFOSE | UJ |
| WL3-WAT-MID-01-091720 | K2008582-006 | PFDS | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | NEtFOSE | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | PFDS | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | PFTrDA | UJ |

| Field Sample ID | Lab ID | Compounds | Qualifier |
|---|---|---|---|
| WL15A-WAT-MID-01-091620 | K2008582-014 | PFTeDA | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | EtFOSA | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | NMeFOSE | UJ |

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016)<br>WL9-WAT-BULK-091620 (K2008582-017) |
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were qualified as estimated (UJ/J):<br>WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016)<br>WL9-WAT-BULK-091620 (K2008582-017) |
| CCV KC2000537 10/07/20 05:53 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000521 10/01/20 18:00 | 8:2 FTS | 138 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | Results not reported from this CCV. |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | |
| | PFTeDA | 170 %R | 70-130% | |
| | NEtFOSE | 292 %R | 70-130% | |
| | NMeFOSE | 267 %R | 70-130% | |
| | 6:2 FTS | 134 %R | 70-130% | |
| CCV KC2000572 (10/23/20 5:52) 700484 | PFTeDA | 142 %R | 70-130% | Associated results were nondetect. No qualifiers. |
| | EtFOSAA | 185 %R | 70-130% | Results not reported from this CCV. |
| | EtFOSA | 143 %R | 70-130% | |

**Trip blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified B.

| Blank ID | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|
| Trip Blank | PFHpA | 0.78 J | WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016) |
| Trip Blank | PFTeDA | 2.3 J | WL6-WAT-BULK-01-091720 (K2008582-003)<br>WL3-WAT-MID-01-091720 (K2008582-005)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016) |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014249-04 | 9/29/20 9:47 | 6:2 FTS | 4.3 J | WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016)<br>WL9-WAT-BULK-091620 (K2008582-017) |
| | | 8:2 FTS | 0.16 J | Associated results were nondetect. |
| KQ2014290-03 | 9/29/2020 15:17 | PFDoA | 1.4 J | WL6-WAT-BULK-01-091720 (K2008582-004)<br>WL21-WAT-MID-01-091620 (K2008582-010)<br>WL8A-WAT-MID-01-091620 (K2008582-012)<br>WL15A-WAT-MID-01-091620 (K2008582-014) |
| | | MeFOSA | 0.51 J | WL21-WAT-MID-01-091620 (K2008582-010) |
| | | PFTeDA | 2.6 J | No associated results. |
| | | PFTrDA | 3.6 J | |
| KQ2016108-03 | 10/22/2020 8:46 | MeFOSA | 0.62 J | No associated results. |
| | | NEtFOSE | 0.18 J | WL6-WAT-BULK-01-091720 (K2008582-004) |
| | | PFOA | 0.99 J | No associated results. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014505-02 | 10/1/20 18:11 | EtFOSA | 0.60 J | WL15-WAT-MID-01-091620 (K2008582-001) |
| KQ2014704-02 | 10/2/20 14:32 | PFOA | 0.36 J | No associated results. |
| KQ2014730-02 | 10/5/20 21:16 | MeFOSA | 0.77 J | Associated results were nondetect. |
| | | EtFOSA | 0.40 J | |
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | No associated results. |
| | | PFTeDA | 3.1 J | |
| | | MeFOSA | 0.58 J | |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |
| KQ2016454-02 | 10/23/20 06:03 | PFOS | 0.57 J | No associated results. |
| | | PFOA | 0.62 J | |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Three results were rejected (R) due to severely low recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL15-WAT-MID-01-091620 | K2008582-002 | 13C2-PFDoDA | 44 | UJ |
| | | 13C2-PFTeDA | 22 | UJ |
| | | D3-MeFOSAA | 29 | UJ |
| | | D5-EtFOSAA | 24 | UJ |
| | | D9-EtFOSE | 47 | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-003 | 13C2-4:2 FTS | 327 | No Qualifier |
| | | 13C2-6:2 FTS | 238 | No Qualifier |
| | | 13C2-PFTeDA | 45 | J+ |
| | | D9-EtFOSE | 40 | UJ |
| WL6-WAT-BULK-01-091720 | K2008582-004 | 13C2-PFDoDA | 33 | J+ |
| | | 13C2-PFTeDA | 21 | UJ |
| | | D3-MeFOSA | 46 | UJ |
| | | D3-MeFOSAA | 26 | UJ |
| | | D5-EtFOSAA | 22 | UJ |
| WL3-WAT-MID-01-091720 | K2008582-005 | 13C2-4:2 FTS | 298 | No Qualifier |
| | | 13C2-6:2 FTS | 237 | No Qualifier |
| | | D9-EtFOSE | 47 | J+ |
| WL3-WAT-MID-01-091720 | K2008582-006 | 13C2-PFDoDA | 38 | UJ |
| | | 13C2-PFTeDA | 34 | UJ |
| | | D3-MeFOSAA | 32 | UJ |
| | | D5-EtFOSAA | 26 | UJ |
| WL14-WAT-MID-01-091620 | K2008582-008 | 13C2-PFDoDA | 40 | UJ |
| | | 13C2-PFTeDA | 23 | UJ |
| | | D3-MeFOSAA | 29 | UJ |
| | | D5-EtFOSAA | 26 | UJ |
| WL21-WAT-MID-01-091620 | K2008582-009 | 13C2-PFTeDA | 44 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL21-WAT-MID-01-091620 | K2008582-010 | 13C2-PFDoDA | 34 | J+ |
| | | 13C2-PFTeDA | 21 | UJ |
| | | 13C2-PFUnDA | 49 | UJ |
| | | 13C8-FOSA | 34 | J+ |
| | | D3-MeFOSA | 9 | R |
| | | D3-MeFOSAA | 12 | R |
| | | D5-EtFOSAA | 15 | R |
| WL8A-WAT-MID-01-091620 | K2008582-011 | 13C2-PFTeDA | 46 | J+ |
| WL8A-WAT-MID-01-091620 | K2008582-012 | 13C2-PFDoDA | 32 | J+ |
| | | 13C2-PFTeDA | 28 | UJ |
| | | D3-MeFOSA | 41 | UJ |
| | | D3-MeFOSAA | 24 | UJ |
| | | D5-EtFOSAA | 20 | UJ |
| WL15A-WAT-MID-01-091620 | K2008582-014 | 13C2-PFDoDA | 40 | J+ |
| | | 13C2-PFTeDA | 25 | UJ |
| | | D3-MeFOSA | 34 | UJ |
| | | D3-MeFOSAA | 39 | UJ |
| | | D5-EtFOSAA | 21 | J+ |
| WL9-WAT-BULK-091620 | K2008582-017 | 13C2-PFTeDA | 48 | UJ |
| TRIP BLANK | K2008582-018 | D9-EtFOSE | 44 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ) and detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014290-01/ KQ2014290-02 (698190) | 11Cl-PF3OUdS | 73/**68** | 70-130 | Associated result results were nondetect and qualified UJ:<br>WL15-WAT-MID-01-091620 (K2008582-002)<br>WL6-WAT-BULK-01-091720 (K2008582-004)<br>WL3-WAT-MID-01-091720 (K2008582-006)<br>WL14-WAT-MID-01-091620 (K2008582-008)<br>WL21-WAT-MID-01-091620 (K2008582-010)<br>WL8A-WAT-MID-01-091620 (K2008582-012)<br>WL15A-WAT-MID-01-091620 (K2008582-014) |
| KQ2014249-03 (698697) | 6:2 FTS | **184/**-- | 70-130 | J+:<br>WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016)<br>WL9-WAT-BULK-091620 (K2008582-017) |
| | 8:2 FTS | **132/**-- | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFNS | **68/**-- | 70-130 | Associated result results were nondetect and |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFDS | **61/**-- | 70-130 | qualified UJ:<br>WL15-WAT-MID-01-091620 (K2008582-001)<br>WL14-WAT-MID-01-091620 (K2008582-007)<br>WL21-WAT-MID-01-091620 (K2008582-009)<br>WL8A-WAT-MID-01-091620 (K2008582-011)<br>WL15A-WAT-MID-01-091620 (K2008582-013)<br>VB3-WAT-BULK-01-091620 (K2008582-015)<br>EP13-WAT-BULK-01-091620 (K2008582-016)<br>WL9-WAT-BULK-091620 (K2008582-017) |
| KQ2014290-01/ KQ2014290-02 (699134) | MeFOSAA | 122**/132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoDA | **146/133** | 70-130 | J+:<br>WL6-WAT-BULK-01-091720 (K2008582-004)<br>WL21-WAT-MID-01-091620 (K2008582-010)<br>WL8A-WAT-MID-01-091620 (K2008582-012)<br>WL15A-WAT-MID-01-091620 (K2008582-014) |
| | PFUnDA | 126/**131** | 70-130 | J+:<br>WL8A-WAT-MID-01-091620 (K2008582-012) |
| | PFPeS | **143/**125 | 70-130 | J+:<br>WL15-WAT-MID-01-091620 (K2008582-002)<br>WL6-WAT-BULK-01-091720 (K2008582-004)<br>WL3-WAT-MID-01-091720 (K2008582-006)<br>WL21-WAT-MID-01-091620 (K2008582-010)<br>WL8A-WAT-MID-01-091620 (K2008582-012)<br>WL14-WAT-MID-01-091620 (K2008582-008)<br>WL15A-WAT-MID-01-091620 (K2008582-014) |
| KQ2014290-01/ KQ2014290-02 (699503) | PFDS | **67/58** | 70-130 | No associated results. |
| | PFTrDA | **148/157** | 70-130 | |
| KQ2016108-01/ KQ2016108-02 (700485) | EtFOSA | **154/145** | 70-130 | Associated results were nondetect. No qualifiers. |
| | NMeFOSE | **137/153** | 70-130 | |
| | PFTeDA | **134/**124 | 70-130 | |
| | NEtFOSE | **187/226** | 70-130 | J+:<br>WL6-WAT-BULK-01-091720 (K2008582-004) |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008583.01 Report date (mm/dd/yyyy): 11/13/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/28/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicated a trip blank was received but was not listed on the COC.<br>Receipt form discusses issues with water samples (2 listed on COC were not received, and two additional samples were received that were not listed on COC). |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | The trip blank detects were less than the RL:<br>PFHpA 1.1 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for summary of exceedances and qualifications. |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/28/2020

## Data Validation Qualifications for K2008583.01

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV KC2000537 10/07/20 05:53 698190 | 11Cl-PF3OUdS | 132 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| CCV KC2000531 (10/6/20 23:25) 698189 | PFBS | 139 %R | 70-130% | Results not reported from this CCV. |
| | PFOA | 145 %R | 70-130% | |
| | PFTrDA | 136 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | NMeFOSE | 267 %R | 70-130% | |
| | PFTeDA | 170 %R | 70-130% | J+: RC6-WAT-BULK-01-091820 (K2008583-002) |
| | NEtFOSE | 292 %R | 70-130% | |
| | 6:2 FTS | 134 %R | 70-130% | |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014429-03 | 10/1/20 7:40 | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | MeFOSA | 0.75 J | |
| | | NMeFOSE | 1.1 J | |
| | | NEtFOSE | 0.93 J | RC6-WAT-BULK-01-091820 (K2008583-002) |
| | | PFTeDA | 3.7 J | |
| | | PFBS | 0.3 J | No associated results. |
| KQ2014435-03 | 10/1/20 9:03 | EtFOSAA | 1.3 J | RC6A-WAT-BULK-01-092020 (K2008583-003) |
| | | NEtFOSE | 0.16 J | RC6-WAT-BULK-01-091820 (K2008583-001) |
| | | PFHpA | 0.96 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFBA | 0.53 J | |
| | | PFHpA | 0.73 J | |
| | | PFTrDA | 1.4 J | Associated results were nondetect. |
| | | MeFOSA | 0.71 J | |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014843-02 | 10/6/20 23:36 | PFHpA | 0.66 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFTeDA | 3.1 J | RC6-WAT-BULK-01-091820 (K2008583-002) |
| | | MeFOSA | 0.58 J | Associated results were nondetect. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | RC6A-WAT-BULK-01-092020 (K2008583-004)<br>RC7-WAT-BULK-02-091920 (K2008583-009)<br>RC7-WAT-BULK-01-091920 (K2008583-008)<br>RC7-WAT-BULK-01-091920 (K2008583-007)<br>RC6A-POWAT-01-092120 (K2008583-005)<br>RC6A-POWAT-02-092120 (K2008583-006)<br>RC22-WAT-BULK-01-091920 (K2008583-011) |
| | | MeFOSA | 0.62 J | RC7-WAT-BULK-02-091920 (K2008583-009) |
| | | EtFOSAA | 0.92 J | RC7-WAT-BULK-01-091920 (K2008583-007)<br>RC7-WAT-BULK-01-091920 (K2008583-008)<br>RC7-WAT-BULK-02-091920 (K2008583-009) |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | Associated results were nondetect. |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC6-WAT-BULK-01-091820 | K2008583-002 | 13C2-4:2 FTS | 207 | No Qualifier |
| | | D9-EtFOSE | 36 | J+ |
| RC6A-WAT-BULK-01-092020 | K2008583-003 | 13C2-4:2 FTS | 233 | No Qualifier |
| RC6A-POWAT-02-092120 | K2008583-006 | 13C3-PFBS | 44 | J+ |
| RC7-WAT-BULK-01-091920 | K2008583-007 | 13C2-4:2 FTS | 207 | No Qualifier |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014429-01/ KQ2014429-02 (698190) | 4:2 FTS | **137/**120 | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **138/**125 | 70-130 | J+: RC6-WAT-BULK-01-091820 (K2008583-002) |
| | PFBA | **134/**130 | 70-130 | |
| KQ2014429-01/ KQ2014429-02 (699503) | PFPeS | **166/153** | 70-130 | J+: RC6-WAT-BULK-01-091820 (K2008583-002) |
| | PFTrDA | **139/**109 | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 120/**178** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFPeS | 119/**145** | 70-130 | J+: RC6A-WAT-BULK-01-092020 (K2008583-004) RC7-WAT-BULK-01-091920 (K2008583-008) RC7-WAT-BULK-02-091920 (K2008583-009) RC7-POWAT-01-092020 (K2008583-010) RC22-WAT-BULK-01-091920 (K2008583-012) |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014524-01/ KQ2014524-02 (698467) | PFPeS | 130/**133** | 70-130 | J+: RC7-WAT-BULK-01-091920 (K2008583-007) |
| KQ2014635-01/ KQ2014635-02 (698705) | PFDoDA | 129/**149** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFUnA | 125/**134** | 70-130 | |
| | PFHpS | **132/136** | 70-130 | J+: RC6A-POWAT-01-092120 (K2008583-005) RC6A-POWAT-02-092120 (K2008583-006) |
| | PFOS | 127/**132** | 70-130 | |
| | PFPeS | **146/141** | 70-130 | |
| KQ2014635-01/ KQ2014635-02 (698706) | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014516-01/ KQ2014516-02 (698467) | 10:2 FTS | 39 | 30 | Associated results were nondetect. No qualifiers. |
| | PFHpS | 35 | 30 | J: RC6A-WAT-BULK-01-092020 (K2008583-004) RC7-WAT-BULK-02-091920 (K2008583-009) RC7-POWAT-01-092020 (K2008583-010) RC22-WAT-BULK-01-091920 (K2008583-012) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008624.01 Report date (mm/dd/yyyy): 11/12/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/29/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | Receipt form indicates the collection date on the COC did not match the sample labels for one sample. The ID on the COC was used as it matched the date in the sample ID.<br>COCs also include water samples not reported in this SDG. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used during review.<br>See data qualification summary for exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for associated samples and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for exceedances and qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS sed/other limits used: 50% to 150%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | KQ2014340-03:<br>10:2 FTS 178%<br>KQ2014341-03:<br>PFPeS 159%<br>Associated results were nondetect. No qualifiers. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | EP4-SED-0-6-03-092420 (K2008624-011)<br>EP27B-SED-0-6-092420 (K2008624-021) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | EP4-SED-0-6-03-092420 (K2008624-011):<br>10:2 FTS 216%/209%<br>EP27B-SED-0-6-092420 (K2008624-021):<br>PFPeS: 176% (MSD %R ok)<br>Associated results were nondetect. No qualifiers. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | MSDs<br>FDs: EP4-SED-0-6-01-092420/EP4-SED-0-6-02-092420/EP4-SED-0-6-03-092420<br>RC13-SED-0-6-01-092320/RC13-SED-0-6-02-092320 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 50% RPD limit for PFAS (sed).<br>MS/MSD RPDs ok<br>See data qualification summary for field duplicate exceedances and qualifications. |

## 7. Method detection limits/Report limits

| **Question** | | **Yes** | **No** | **N/A** | **Comments** |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| **Questions** | | **Yes** | **No** | **N/A** | **Comments** |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| **Question** | | **Yes** | **No** | **N/A** | **Comments** |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2020

## Data Validation Qualifications for K2008624.01

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %D or %R | Limit | Qualifiers |
|---|---|---|---|---|
| ICV KC2000521-08 (9/29/20) | EtFOSAA | 36.4 %D | ±30 %D | Associated results were qualified as estimated (UJ/J):<br><br>WL16-SED-0-6-01-092320 (K2008624-001)<br>WL10A-SED-0-6-01-092220 (K2008624-002)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL8A-SED-0-6-01-092220 (K2008624-004)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>WL7-SED-0-6-01-092420 (K2008624-007)<br>EP5-SED-0-6-01-092420 (K2008624-008)<br>EP4-SED-0-6-01-092420 (K2008624-009)<br>EP4-SED-0-6-02-092420 (K2008624-010)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>WL6-SED-0-6-01-092420 (K2008624-012)<br>WL2-SED-0-6-01-092220 (K2008624-013)<br>RC18A-SED-0-6-01-092320 (K2008624-014)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21A-SED-0-6-01-092220 (K2008624-016)<br>RC21B-SED-0-6-01-092220 (K2008624-017)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020)) |
| ICV KC2000533-08 (9/29/20) | 11Cl-PF3OUdS | 38.22 %D | ±30 %D | Associated results were nondetect and qualified as estimated (UJ):<br><br>WL16-SED-0-6-01-092320 (K2008624-001)<br>WL10A-SED-0-6-01-092220 (K2008624-002)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL8A-SED-0-6-01-092220 (K2008624-004)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>WL7-SED-0-6-01-092420 (K2008624-007)<br>EP5-SED-0-6-01-092420 (K2008624-008)<br>EP4-SED-0-6-01-092420 (K2008624-009)<br>EP4-SED-0-6-02-092420 (K2008624-010)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>WL6-SED-0-6-01-092420 (K2008624-012)<br>WL2-SED-0-6-01-092220 (K2008624-013)<br>RC18A-SED-0-6-01-092320 (K2008624-014)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21A-SED-0-6-01-092220 (K2008624-016)<br>RC21B-SED-0-6-01-092220 (K2008624-017)<br>RC13-SED-0-6-01-092320 (K2008624-018)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020) |
| CCV KC2000521 (10/02/20 21:07) 697957 | 8:2 FTS | 139 %R | 70-130% | Associated results were nondetect and were acceptable without qualification. |
| | 10:2 FTS | 196 %R | 70-130% | |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| Method Blank KQ2014340-04 | 8:2 FTS | 0.03 J | RC14-SED-0-6-01-092320 (K2008624-020) |
| | PFTeDA | 0.25 J | WL16-SED-0-6-01-092320 (K2008624-001)<br>WL10A-SED-0-6-01-092220 (K2008624-002)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL8A-SED-0-6-01-092220 (K2008624-004)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>WL7-SED-0-6-01-092420 (K2008624-007)<br>EP5-SED-0-6-01-092420 (K2008624-008)<br>EP4-SED-0-6-01-092420 (K2008624-009)<br>EP4-SED-0-6-02-092420 (K2008624-010)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>WL6-SED-0-6-01-092420 (K2008624-012)<br>WL2-SED-0-6-01-092220 (K2008624-013)<br>RC18A-SED-0-6-01-092320 (K2008624-014)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21A-SED-0-6-01-092220 (K2008624-016)<br>RC13-SED-0-6-01-092320 (K2008624-018)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020) |
| | MeFOSA | 0.11 J | WL16-SED-0-6-01-092320 (K2008624-001)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21B-SED-0-6-01-092220 (K2008624-017)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020) |
| CCB KQ2014699-02 10/2/20 21:18 | PFTeDA | 0.35 J | WL16-SED-0-6-01-092320 (K2008624-001)<br>WL10A-SED-0-6-01-092220 (K2008624-002)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL8A-SED-0-6-01-092220 (K2008624-004)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>WL7-SED-0-6-01-092420 (K2008624-007)<br>EP5-SED-0-6-01-092420 (K2008624-008)<br>EP4-SED-0-6-01-092420 (K2008624-009)<br>EP4-SED-0-6-02-092420 (K2008624-010)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>WL6-SED-0-6-01-092420 (K2008624-012)<br>WL2-SED-0-6-01-092220 (K2008624-013)<br>RC18A-SED-0-6-01-092320 (K2008624-014)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21A-SED-0-6-01-092220 (K2008624-016)<br>RC13-SED-0-6-01-092320 (K2008624-018)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020) |
| | EtFOSA | 0.16 J | Associated results were nondetect. No qualifiers. |

| Blank Sample ID | Analyte | Concentration (ng/g) | Results Qualified B |
|---|---|---|---|
| CCB KQ2015283-02 10/11/20 23:14 | MeFOSA | 0.25 J | WL16-SED-0-6-01-092320 (K2008624-001)<br>WL13-SED-0-6-01-092520 (K2008624-003)<br>WL11-SED-0-6-01-092220 (K2008624-005)<br>WL10-SED-0-6-01-092220 (K2008624-006)<br>EP4-SED-0-6-03-092420 (K2008624-011)<br>RC17A-SED-0-6-01-092320 (K2008624-015)<br>RC21B-SED-0-6-01-092220 (K2008624-017)<br>RC13-SED-0-6-02-092320 (K2008624-019)<br>RC14-SED-0-6-01-092320 (K2008624-020) |
| CCB KQ2015285-02 10/12/20 06:30 | EtFOSA | 0.15 J | Associated results were nondetect. No qualifiers. |
| | MeFOSA | 0.18 J | EP27B-SED-0-6-092420 (K2008624-021) |
| | EtFOSAA | 0.24 J | RC13-SED-0-6-01-092320 (K2008624-018)<br>EP27B-SED-0-6-092420 (K2008624-021) |
| CCB KQ2015731-02 10/15/20 08:45 | MeFOSA | 0.086 J | Associated results were nondetect. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL10A-SED-0-6-01-092220 | K2008624-002 | 13C3-PFBS | 42 | UJ |
| WL6-SED-0-6-01-092420 | K2008624-012 | 13C3-PFBS | 45 | UJ |
| RC13-SED-0-6-01-092320 | K2008624-018 | 13C3-PFBS | 46 | UJ |
| RC13-SED-0-6-02-092320 | K2008624-019 | 13C3-PFBS | 42 | UJ |
| RC14-SED-0-6-01-092320 | K2008624-020 | 13C3-PFBS | 48 | UJ |

**Field duplicate results (Section 6Bi):** Detects associated with high RPD were qualified as estimated (J):

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **EP4-SED-0-6-01-092420/EP4-SED-0-6-02-092420/EP4-SED-0-6-03-092420:** | | | | |
| PFOS | 4.9 | 2.7 | 4.1 | 56 |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008630 Report date (mm/dd/yyyy): 10/28/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/29/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A number of sediment samples are listed on the COCs that were not reported in this SDG.<br>Receipt form discusses filter/unfilter labeling issues for sample WL11-POWAT-01-092220. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects. No qualifiers. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs<br>FD: WL8A-POWAT-01-092220-F/WL8A-POWAT-02-092220-F/WL8A-POWAT-03-092220-F |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for summary of exceedances and qualifications.<br>FD (30%RPD or ±RL): ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2020

## Data Validation Qualifications for K2008630

**Calibration (Section 2):** Calibration noncompliances are summarized below.

| Calibration (Date) Batch | Analyte | %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV KC2000572 (10/26/20 15:44) 700785 | PFHxS | 136 %R | 70-130% | Results not reported from this CCV. |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014746-03 | 10/7/2020 7:55 | PFBA | 0.42 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFHpA | 0.63 J | |
| KQ2014877-03 | 10/7/2020 7:56 | PFBA | 0.53 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2015031-02 | 10/8/20 10:29 | PFHpA | 0.67 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | MeFOSA | 0.62 J | Associated results were nondetect. |
| | | EtFOSAA | 0.92 J | WL11-WAT-BULK-01-092220 (K2008630-011) |
| KQ2015034-02 | 10/8/20 21:12 | MeFOSA | 0.63 J | Associated results were nondetect. |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | EtFOSE | 0.18 J | Associated results were nondetect. |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | Associated results were nondetect. |
| KQ2016092-02 | 10/20/20 13:59 | PFOA | 0.37 J | No associated results. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were nondetect and qualified as estimated (UJ).

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL16-WAT-BULK-01-092320 | K2008630-002 | 13C2-PFTeDA | 47 | UJ |
| WL16-POWAT-01-092320 | K2008630-003 | 13C2-PFTeDA | 48 | UJ |
| WL13-POWAT-01-092520 | K2008630-004 | 13C2-PFTeDA | 35 | UJ |
| WL10A-POWAT-01-092220 | K2008630-006 | 13C2-PFTeDA | 46 | UJ |
| WL8A-POWAT-01-092220 | K2008630-007 | 13C2-PFTeDA | 43 | UJ |
| WL8A-POWAT-03-092220 | K2008630-009 | 13C2-PFTeDA | 40 | UJ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| WL11-WAT-BULK-01-092220 | K2008630-011 | 13C2-4:2 FTS | 207 | No Qualifier |
| WL11-WAT-BULK-01-092220 | K2008630-012 | 13C2-PFTeDA | 41 | UJ |
| WL11-WAT-BULK-01-092220 | K2008630-012 | D3-MeFOSAA | 45 | UJ |
| WL7-POWAT-01-092420 | K2008630-013 | 13C2-4:2 FTS | 217 | No Qualifier |
| WL7-POWAT-01-092420 | K2008630-013 | 13C2-PFTeDA | 44 | UJ |
| WL7-POWAT-01-092420 | K2008630-014 | 13C2-PFTeDA | 45 | UJ |
| WL6-POWAT-01-092420 | K2008630-015 | 13C2-PFTeDA | 46 | UJ |
| WL6-POWAT-01-092420 | K2008630-016 | 13C2-PFTeDA | 46 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014524-01/ KQ2014524-02 (698467) | PFPeS | 130/**133** | 70-130 | J+: WL16-WAT-BULK-01-092320 (K2008630-001) WL11-WAT-BULK-01-092220 (K2008630-011) |
| KQ2014635-01/ KQ2014635-02 (698705) | PFDoDA | 129/**149** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFUnA | 125/**134** | 70-130 | |
| | PFOS | 127/**132** | 70-130 | J+: WL16-WAT-BULK-01-092320 (K2008630-002) WL8A-POWAT-01-092220 (K2008630-007) WL8A-POWAT-02-092220 (K2008630-008) WL8A-POWAT-03-092220 (K2008630-009) WL11-POWAT-01-092220 (K2008630-010) WL11-WAT-BULK-01-092220 (K2008630-012) |
| | PFHpS | **132/136** | 70-130 | |
| | PFPeS | **146/141** | 70-130 | |
| KQ2014635-01/ KQ2014635-02 (698706) | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698706) | 11Cl-PF3OUdS | **132/146** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698705) | 8:2 FTS | **138/133** | 70-130 | Associated results were nondetect. No qualifiers. |
| | EtFOSAA | 101/**133** | 70-130 | |
| | PFDoA | **138/143** | 70-130 | |
| | PFNS | 123/**133** | 70-130 | |
| | PFUnA | 125/**141** | 70-130 | |
| | PFOS | 125/**132** | 70-130 | J+: WL13-POWAT-01-092520 (K2008630-005) WL7-POWAT-01-092420 (K2008630-014) WL6-POWAT-01-092420 (K2008630-016) |
| | PFPeS | **164/163** | 70-130 | |
| KQ2014783-01/ KQ2014783-02 (698706) | 11Cl-PF3OUdS | **144/149** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014783-01/ KQ2014783-02 | 8:2 FTS | 124/**142** | 70-130 | Associated results were nondetect. No qualifiers. |
| | EtFOSAA | 106/**135** | 70-130 | |
| | PFDoA | **133/141** | 70-130 | |
| | PFNS | 124/**134** | 70-130 | |

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFUnA | **135/143** | 70-130 | |
| | PFOS | **131/**110 | 70-130 | J+: |
| | PFPeS | **155/159** | 70-130 | WL16-POWAT-01-092320 (K2008630-003)<br>WL10A-POWAT-01-092220 (K2008630-006) |
| KQ2014877-01/ KQ2014877-02 (698780) | 11Cl-PF3OUdS | **138/147** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014877-01/ KQ2014877-02 (698470) | PFNS | **136/139** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFHxS | 119**/137** | 70-130 | J+: |
| | PFPeS | **135/**114 | 70-130 | WL13-POWAT-01-092520 (K2008630-004)<br>WL7-POWAT-01-092420 (K2008630-013)<br>WL6-POWAT-01-092420 (K2008630-015) |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014877-01/ KQ2014877-02 (698780) | PFHpS | 41 | 30 | J+:<br>WL13-POWAT-01-092520 (K2008630-004)<br>WL7-POWAT-01-092420 (K2008630-013)<br>WL6-POWAT-01-092420 (K2008630-015) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008635 Report date (mm/dd/yyyy): 10/27/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/29/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A number of sediment samples are listed on the COCs that were not reported in this SDG. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used for review. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualification summary for summary of detects. No qualifiers. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for calibration blank detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for summary of exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2020

## Data Validation Qualifications for K2008635

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014746-03 | 10/7/2020 7:55 | PFBA | 0.42 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFHpA | 0.63 J | |
| KQ2014877-03 | 10/7/2020 7:56 | PFBA | 0.53 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2015034-02 | 10/8/20 21:12 | MeFOSA | 0.63 J | EP5-WAT-BULK-01-092420 (K2008635-003) |
| | | EtFOSA | 0.38 J | Associated results were nondetect. |
| | | EtFOSE | 0.18 J | EP5-WAT-BULK-01-092420 (K2008635-003)<br>EP4-WAT-BULK-01-092420 (K2008635-006) |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | EP5-WAT-BULK-01-092420 (K2008635-004) |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| EP23-WAT-BULK-01-092420 | K2008635-001 | 13C2-PFTeDA | 29 | J+ |
| EP23-WAT-BULK-01-092420 | K2008635-002 | 13C2-PFTeDA | 39 | UJ |
| EP5-WAT-BULK-01-092420 | K2008635-003 | 13C2-4:2 FTS | 230 | No Qualifier |
| EP5-WAT-BULK-01-092420 | K2008635-003 | 13C2-PFTeDA | 31 | J+ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | 13C2-PFTeDA | 32 | UJ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | 13C8-FOSA | 24 | J+ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D3-MeFOSA | 34 | J+ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D3-MeFOSAA | 38 | UJ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D5-EfFOSAA | 43 | UJ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D5-EtFOSA | 27 | UJ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D7-MeFOSE | 27 | J+ |
| EP5-WAT-BULK-01-092420 | K2008635-004 | D9-EtFOSE | 30 | UJ |
| EP4-WAT-BULK-01-092420 | K2008635-006 | 13C2-PFTeDA | 26 | J+ |
| EP4-WAT-BULK-01-092420 | K2008635-007 | 13C2-PFTeDA | 33 | UJ |
| EP4-WAT-BULK-01-092420 | K2008635-007 | D3-MeFOSAA | 42 | UJ |
| EP4-WAT-BULK-01-092420 | K2008635-007 | D5-EfFOSAA | 49 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014635-01/ KQ2014635-02 (698705) | PFDoDA | 129/**149** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFUnA | 125/**134** | 70-130 | |
| | PFHpS | **132/136** | 70-130 | |
| | PFOS | 127/**132** | 70-130 | J+: WL2-POWAT-01-092220 (K2008635-009) |
| | PFPeS | **146/141** | 70-130 | |
| KQ2014635-01/ KQ2014635-02 (698706) | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698706) | 11Cl-PF3OUdS | **132/146** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698705) | 8:2 FTS | **138/133** | 70-130 | J+: EP4-WAT-BULK-01-092420 (K2008635-007) |
| | EtFOSAA | 101/**133** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoA | **138/143** | 70-130 | |
| | PFNS | 123/**133** | 70-130 | |
| | PFUnA | 125/**141** | 70-130 | |
| | PFOS | 125/**132** | 70-130 | J+: EP23-WAT-BULK-01-092420 (K2008635-002) EP5-WAT-BULK-01-092420 (K2008635-004) EP5-POWAT-01-092420 (K2008635-005) EP4-WAT-BULK-01-092420 (K2008635-007) EP4-POWAT-01-092420 (K2008635-008) |
| | PFPeS | **164/163** | 70-130 | |
| KQ2014877-01/ KQ2014877-02 (698780) | 11Cl-PF3OUdS | **138/147** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014877-01/ KQ2014877-02 (698470) | PFNS | **136/139** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFHxS | 119/**137** | 70-130 | J+: EP23-WAT-BULK-01-092420 (K2008635-001) EP5-WAT-BULK-01-092420 (K2008635-003) EP4-WAT-BULK-01-092420 (K2008635-006) |
| | PFPeS | **135/**114 | 70-130 | |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014877-01/ KQ2014877-02 (698780) | PFHpS | 41 | 30 | J: EP23-WAT-BULK-01-092420 (K2008635-001) EP5-WAT-BULK-01-092420 (K2008635-003) EP4-WAT-BULK-01-092420 (K2008635-006) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008636 Report date (mm/dd/yyyy): 10/26/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/29/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A number of sediment samples are listed on the COCs that were not reported in this SDG. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used for review. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☐ | ☒ | ☐ | |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☐ | ☒ | |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☐ | ☐ | ☒ | |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☐ | ☒ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☐ | ☒ | ☐ | See data qualification summary for summary of detects. No qualifiers. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects. No qualifiers. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%. |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☐ | ☒ | ☐ | None for this SDG. |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☒ | ☐ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☐ | ☒ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water).<br>LCSD - See data qualification summary for summary of exceedances and qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☐ | ☐ | ☒ | |
| **C.** | Are percent moisture results reported? | ☐ | ☐ | ☒ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2020

## Data Validation Qualifications for K2008636

**Method blank and instrument blank detections (Section 3Bii and C):** Detection in the associated blanks are listed below. Results did not require qualification.

| Method Blank ID | Prep Date | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2014746-03 | 10/7/2020 7:55 | PFBA | 0.42 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFHpA | 0.63 J | Associated results were nondetect or greater than 10 times the blank concentration. No qualifiers. |
| KQ2014877-03 | 10/7/2020 7:56 | PFBA | 0.53 J | Associated results were greater than 10 times the blank concentration. No qualifiers. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration (ng/L) | Results Qualified B |
|---|---|---|---|---|
| KQ2015034-02 | 10/8/20 21:12 | MeFOSA | 0.63 J | Associated results were nondetect. |
| | | EtFOSA | 0.38 J | |
| | | EtFOSE | 0.18 J | |
| KQ2015208-02 | 10/9/20 15:39 | MeFOSA | 0.68 J | Associated results were nondetect. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC13-POWAT-01-092320 | K2008636-004 | 13C2-PFTeDA | 34 | UJ |
| RC13-POWAT-01-092320 | K2008636-004 | D3-MeFOSAA | 39 | UJ |
| RC13-POWAT-01-092320 | K2008636-004 | D5-EfFOSAA | 48 | UJ |
| RC14-POWAT-01-092320 | K2008636-005 | 13C2-PFTeDA | 47 | UJ |
| EP27B-WAT-BULK-01-092420 | K2008636-006 | 13C2-4:2 FTS | 223 | No Qualifier |
| EP27B-WAT-BULK-01-092420 | K2008636-006 | 13C2-PFTeDA | 31 | J+ |
| EP27B-WAT-BULK-01-092420 | K2008636-007 | 13C2-PFTeDA | 36 | UJ |
| EP27B-POWAT-01-092420 | K2008636-008 | 13C2-PFTeDA | 32 | UJ |
| EP27B-POWAT-01-092420 | K2008636-008 | D5-EfFOSAA | 48 | UJ |
| EP17C-WAT-BULK-01-092420 | K2008636-009 | 13C2-4:2 FTS | 210 | UJ |
| EP17C-WAT-BULK-01-092420 | K2008636-009 | 13C2-PFTeDA | 40 | J+ |
| EP17C-WAT-BULK-01-092420 | K2008636-010 | 13C2-PFTeDA | 37 | UJ |
| EP17C-WAT-BULK-01-092420 | K2008636-010 | D3-MeFOSAA | 46 | UJ |
| EP17C-WAT-BULK-01-092420 | K2008636-010 | D5-EfFOSAA | 47 | UJ |
| EP17C-POWAT-01-092420 | K2008636-011 | 13C2-PFTeDA | 48 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (Batch) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014635-01/ KQ2014635-02 (698705) | PFDoDA | 129/**149** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFUnA | 125/**134** | 70-130 | |
| | PFPeS | **146/141** | 70-130 | |
| | PFHpS | **132/136** | 70-130 | |
| | PFOS | 127/**132** | 70-130 | J+:<br>RC21A-POWAT-01-092220 (K2008636-002)<br>RC17A-POWAT-01-092320 (K2008636-003) |
| KQ2014635-01/ KQ2014635-02 (698706) | 11Cl-PF3OUdS | **143/142** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698706) | 11Cl-PF3OUdS | **132/146** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014746-01/ KQ2014746-02 (698705) | 8:2 FTS | **138/133** | 70-130 | Associated results were nondetect. No qualifiers. |
| | EtFOSAA | 101/**133** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFDoA | **138/143** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFNS | 123/**133** | 70-130 | J+:<br>EP27B-POWAT-01-092420 (K2008636-008) |
| | PFUnA | 125/**141** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFOS | 125/**132** | 70-130 | J+:<br>RC18A-POWAT-01-092320 (K2008636-001)<br>RC13-POWAT-01-092320 (K2008636-004)<br>RC14-POWAT-01-092320 (K2008636-005)<br>EP27B-WAT-BULK-01-092420 (K2008636-007)<br>EP27B-POWAT-01-092420 (K2008636-008)<br>EP17C-WAT-BULK-01-092420 (K2008636-010)<br>EP17C-POWAT-01-092420 (K2008636-011) |
| | PFPeS | **164/163** | 70-130 | J+:<br>EP27B-WAT-BULK-01-092420 (K2008636-007)<br>EP27B-POWAT-01-092420 (K2008636-008)<br>EP17C-WAT-BULK-01-092420 (K2008636-010)<br>EP17C-POWAT-01-092420 (K2008636-011) |
| KQ2014877-01/ KQ2014877-02 (698780) | 11Cl-PF3OUdS | **138/147** | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2014877-01/ KQ2014877-02 (698470) | PFNS | **136/139** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFHxS | 119/**137** | 70-130 | J+:<br>EP27B-WAT-BULK-01-092420 (K2008636-006)<br>EP17C-WAT-BULK-01-092420 (K2008636-009) |
| | PFPeS | **135/**114 | 70-130 | |

**Laboratory control sample/Laboratory control sample duplicate RPDs (Section 6Bi):** Detects associated with high RPDs were qualified as J:

| LCS/LCSD IDs (Batch) | Compound | RPD | RPD Limit | Qualifiers |
|---|---|---|---|---|
| KQ2014877-01/ KQ2014877-02 (698780) | PFHpS | 41 | 30 | J:<br>EP27B-WAT-BULK-01-092420 (K2008636-006)<br>EP17C-WAT-BULK-01-092420 (K2008636-009) |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2008909 Report date (mm/dd/yyyy): 10/30/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/29/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A trip blank was recieved; however, was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for summary of exceedances and qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | Trip blank reported |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Trip blank detects were less than the RL:<br>PFBA 0.86 J ng/L<br>PFOA 0.41 J ng/L<br>6:2 FTS 0.95 J ng/L |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>soil/sed limits used: 50% to150% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSD: RC23-SED-0-6-01-092820 (K2008909-003) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs, MSD |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair? If no, explain possible causes and data impact. | | ☒ | ☐ | ☐ | 30% limit for PFAS (water)<br>50% limit for PFAS (Soil/Sed) |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | **Other RPDs may be acceptable. Check with regulatory agency.* | | | | |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/29/2020

## Data Validation Qualifications for K2008909

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV KC2000572 (10/23/20 05:52) | PFTeDA | 142 | 70-130% | Associated detects were qualified as estimated biased high (J+): VAPI-GW-24-28-01-093020 (K2008909-011) |
| | EtFOSA | 143 | 70-130% | Associated results were nondetect. No qualifiers. |
| | EfFOSAA | 185 | 70-130% | Associated detects were qualified as estimated biased high (J+): RC23-WAT-BULK-01-092820 (K2008909-001) |
| CCV KC2000572 (10/26/20 17:52) | MeFOSAA | 131 | 70-130% | Associated results were nondetect. No qualifiers. |

**Trip blank detections (Section 3Aii):** The PFOA trip blank detection was determined to be laboratory contamination per qualifications in the next section.

| Compound | Blank Concentration | Results Qualified B |
|---|---|---|
| PFBA | 0.89 J ng/l | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| 6:2 FTS | 0.95 J ng/l | VAPC-GW-22-26-01-100220 (K2008909-025) |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2014341-04 | 10/8/20 9:55 | MeFOSA | 0.11 J ng/g | RC23-SED-0-6-01-092820 (K2008909-003)<br>VAPH-SOIL-8-12-01-092920 (K2008909-004)<br>VAPH-SOIL-24-27-01-092920 (K2008909-006)<br>VAPI-SOIL-COMP-24-28-01-093020 (K2008909-013)<br>VAPI-SOIL-COMP-36-39-01-093020 (K2008909-014)<br>VAPC-SOIL-COMP-1-5-01-100220 (K2008909-022)<br>VAPC-SOIL-COMP-12-16-01-100220 (K2008909-024) |
| KQ2015280-03 | 10/12/20 8:28 | 6:2 FTS | 0.79 J ng/l | Associated results were nondetect. No qualifiers. |
| | | MeFOSA | 0.63 J ng/l | RC23-WAT-BULK-01-092820 (K2008909-002) |
| | | PFOA | 0.82 J ng/l | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| KQ2015282-04 | 10/12/20 8:30 | EtFOSAA | 1.1 J ng/l | Associated results were nondetect or greater than 10 times the blank concentration. No qualifiers. |
| | | PFBA | 0.42 J ng/l | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFOA | 0.52 J ng/l | VAPI-GW-12-16-01-093020 (K2008909-010)<br>VAPA-GW-6-10-01-100120 (K2008909-017)<br>VAPC-GW-12-16-01-100220 (K2008909-023)<br>TRIP BLANK (K2008909-028) |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2015285-02 | 10/12/20 06:30 | MeFOSA | 0.18 J ng/g | RC23-SED-0-6-01-092820 (K2008909-003)<br>VAPH-SOIL-8-12-01-092920 (K2008909-004)<br>VAPH-SOIL-24-27-01-092920 (K2008909-006)<br>VAPI-SOIL-COMP-24-28-01-093020 (K2008909-013)<br>VAPI-SOIL-COMP-36-39-01-093020 (K2008909-014)<br>VAPC-SOIL-COMP-1-5-01-100220 (K2008909-022)<br>VAPC-SOIL-COMP-12-16-01-100220 (K2008909-024) |
| | | EtFOSA | 0.15 J ng/g | Associated results were nondetect. No qualifiers. |
| | | EtFOSAA | 0.24 J ng/g | Associated results were nondetect or greater than 10 times the blank concentration. No qualifiers. |
| KQ2016535-02 | 10/23/20 06:03 | PFOS | 0.57 J ng/L | VAPI-GW-12-16-01-093020 (K2008909-010)<br>VAPA-GW-6-10-01-100120 (K2008909-017) |
| | | PFOA | 0.62 J ng/L | |
| | | EtFOSAA | 1.2 J ng/L | Associated results were nondetect or greater than 10 times the blank concentration. No qualifiers. |
| KQ2016537-02 | 10/26/20 18:03 | PFOA | 0.51 J ng/L | VAPC-GW-12-16-01-100220 (K2008909-023)<br>TRIP BLANK (K2008909-028) |
| | | MeFOSA | 0.52 J ng/L | RC23-WAT-BULK-01-092820 (K2008909-002)<br>VAPC-GW-12-16-01-100220 (K2008909-023) |
| KQ2016606-02 | 10/27/20 10:59 | PFOA | 0.45 J ng/L | Associated results were greater than 10 times the blank concentration. No qualifiers. |

**Labeled Analogs - surrogates (Section 4Bii):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Eighteen result were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| RC23-WAT-BULK-01-092820 | K2008909-001 | 13C2-PFTeDA | 25 | UJ |
| RC23-WAT-BULK-01-092820 | K2008909-001 | 13C4-PFOS | 48 | UJ:<br>11Cl-PF3OUdS<br>9Cl-PF3ONS<br>ADONA |
| RC23-WAT-BULK-01-092820 | K2008909-002 | 13C2-4:2 FTS | 279 | No Qualifier |
| RC23-WAT-BULK-01-092820 | K2008909-002 | 13C2-6:2 FTS | 232 | No Qualifier |
| RC23-WAT-BULK-01-092820 | K2008909-002 | 13C2-PFTeDA | 47 | UJ |
| VAPH-GW-8-12-01-092920 | K2008909-005 | 13C2-PFTeDA | 23 | UJ |
| VAPH-GW-8-12-01-092920 | K2008909-005 | D3-MeFOSAA | 48 | UJ |
| VAPH-GW-8-12-01-092920 | K2008909-005 | D5-EtFOSAA | 48 | UJ |
| VAPH-GW-20-24-01-092920 | K2008909-007 | 13C8-FOSA | 32 | UJ |
| VAPH-GW-27-30-01-092920 | K2008909-008 | 13C2-PFDoDA | 49 | UJ |
| VAPH-GW-27-30-01-092920 | K2008909-008 | 13C2-PFTeDA | 21 | UJ |
| VAPH-GW-27-30-01-092920 | K2008909-008 | D3-MeFOSAA | 30 | UJ |
| VAPH-GW-27-30-01-092920 | K2008909-008 | D5-EtFOSAA | 29 | UJ |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-6:2 FTS | 25 | UJ |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-8:2 FTS | 2 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-PFDA | 1 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-PFDoDA | 1 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-PFTeDA | 3 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C2-PFUnDA | 1 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C4-PFHpA | 40 | J+ |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C4-PFOA | 12 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C4-PFOS | 1 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C4-PFOA | 10 | R: 11Cl-PF3OUdS 9Cl-PF3ONS ADONA |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C4-PFOS | 1 | |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C5-PFNA | 3 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 13C8-FOSA | 25 | J+ |
| VAPI-GW-24-28-01-093020 | K2008909-011 | 18O2-PFHxS | 18 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D3-MeFOSA | 7 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D3-MeFOSAA | 1 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D5-EtFOSA | 6 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D5-EtFOSAA | 1 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D7-MeFOSE | 8 | R |
| VAPI-GW-24-28-01-093020 | K2008909-011 | D9-EtFOSE | 6 | R |
| VAPA-GW-6-10-01-100120 | K2008909-017 | 13C2-PFTeDA | 23 | UJ |
| VAPA-GW-48-52-01-100120 | K2008909-020 | 13C2-PFTeDA | 38 | UJ |
| VAPA-GW-48-52-01-100120 | K2008909-020 | D3-MeFOSAA | 45 | UJ |
| VAPA-GW-48-52-01-100120 | K2008909-020 | D5-EtFOSAA | 47 | UJ |
| VAPC-GW-0-5-01-100220 | K2008909-021 | 13C2-PFTeDA | 48 | UJ |
| VAPC-GW-22-26-01-100220 | K2008909-025 | 13C2-PFTeDA | 32 | UJ |
| VAPC-GW-43-47-01-100220 | K2008909-026 | 13C2-4:2 FTS | 244 | No Qualifier |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (matrix) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2014341-03 (soil/sed) | PFPeS | **159/**-- | 50-150 | J+: RC23-SED-0-6-01-092820 (K2008909-003) |
| KQ2015280-02 (waters) | PFHpS | 105/**141** | 70-130 | J+: RC23-WAT-BULK-01-092820 (K2008909-002) |
| KQ2015282-03 (waters) | PFHpS | **146/**-- | 70-130 | J+: RC23-WAT-BULK-01-092820 (K2008909-001) VAPH-GW-8-12-01-092920 (K2008909-005) VAPH-GW-20-24-01-092920 (K2008909-007) VAPH-GW-27-30-01-092920 (K2008909-008) VAPI-GW-24-28-01-093020 (K2008909-011) VAPI-GW-36-39-01-093020 (K2008909-015) VAPA-GW-22-26-01-100120 (K2008909-018) VAPA-GW-48-52-01-100120 (K2008909-020) VAPC-GW-0-5-01-100220 (K2008909-021) VAPC-GW-22-26-01-100220 (K2008909-025) VAPC-GW-43-47-01-100220 (K2008909-026) |

| LCS/LCSD IDs (matrix) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| | PFHxS | **132** | 70-130 | J+:<br>RC23-WAT-BULK-01-092820 (K2008909-001)<br>VAPH-GW-8-12-01-092920 (K2008909-005)<br>VAPH-GW-20-24-01-092920 (K2008909-007)<br>VAPH-GW-27-30-01-092920 (K2008909-008)<br>VAPI-GW-24-28-01-093020 (K2008909-011)<br>VAPI-GW-36-39-01-093020 (K2008909-015)<br>VAPA-GW-6-10-01-100120 (K2008909-017)<br>VAPA-GW-22-26-01-100120 (K2008909-018)<br>VAPA-GW-48-52-01-100120 (K2008909-020)<br>VAPC-GW-0-5-01-100220 (K2008909-021)<br>VAPC-GW-22-26-01-100220 (K2008909-025)<br>VAPC-GW-43-47-01-100220 (K2008909-026) |

**Matrix spike/matrix spike sample duplicate (Section 6Av):** Detects associated with high recoveries were qualified as J+ as summarized below:

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **RC23-SED-0-6-01-092820:** | | | |
| 11Cl-PF3OUdS | **151/172** | 50-150 | The result for sample RC23-SED-0-6-01-092820 was nondetect and was acceptable without qualification. |
| PFDoA | 131**/168** | 50-150 | The detects for this sample were qualified as estimated biased high (J+):<br><br>RC23-SED-0-6-01-092820 (K2008909-003) |
| PFNS | 141**/167** | 50-150 | |
| PFPeS | **188/167** | 50-150 | |
| PFUnA | 135/**163** | 50-150 | |
| PFOS | **-156/106** | 50-150 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2009120 Report date (mm/dd/yyyy): 10/30/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/30/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A trip blank was recieved; however, was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for summary of exceedances. No qualifiers. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | One trip blank reported and three equipment blanks (EQ) reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. Seventeen results were rejected (R) due to severely low labeled analog recoveries. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>soil/sed limits used: 50% to 150% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSDs: VAPE-GW-25-29-03-100520 (K2009120-006)<br>VAPE-SOIL-COMP-25-29-03-100520 (K2009120-009 ) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSDs |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs, MSDs<br>FDs: VAPE-GW-25-29-01-100520/VAPE-GW-25-29-02-100520/VAPE-GW-25-29-03-100520<br>VAPE-SOIL-COMP-25-29-01-100520/VAPE-SOIL-COMP-25-29-02-100520/VAPE-SOIL-COMP-25-29-03-100520<br>VAPF-GW-26-30-01-100620/VAPF-GW-26-30-02-100620 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | VAPF-SOIL-COMP-26-30-01-100620/VAPF-SOIL-COMP-26-30-02-100620 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water)<br>50% limit for PFAS (Soil/Sed)<br>LCSD, MSD and FD RPDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | Numerous issues discussed in case narrative. |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

**Seventeen results were rejected (R) due to severely low labeled analog recoveries.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/30/2020

## Data Validation Qualifications for K2009120

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV KC2000572 (10/23/20 05:52) | PFTeDA | 142 | 70-130% | Associated results were nondetect. No qualifiers. |
| | EtFOSA | 143 | 70-130% | |
| | EfFOSAA | 185 | 70-130% | |
| CCV KC2000572 (10/26/20 17:52) | MeFOSAA | 131 | 70-130% | Associated results were nondetect. No qualifiers. |
| CCV KC2000572 (10/29/20 10:35) | NEtFOSE | 144 | 70-130% | Associated results were nondetect. No qualifiers. |

**Trip blank and equipment blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified B.

| Blank ID | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|
| TRIP BLANK | PFOA | 0.39 J ng/l | Laboratory contamination (see method blank qualifications). |
| EQ-PAIL-01-100720 | PFOA | 0.41 J ng/l | Laboratory contamination (see method blank qualifications). |
| EQ-AUGUR-01-100720 | PFOA | 0.57 J ng/l | Laboratory contamination (see method blank qualifications). |
| | PFOS | 0.87 J ng/l | Associated results (RC23-SED-0-6-01-092820) were greater than 10 times the blank concentration. No qualifiers. |
| | PFBA | 0.41 J ng/l | The associated sample result (RC23-SED-0-6-01-092820 reported in SDG K2008909) qualified B. |
| EQ-TUBE-01-100720 | PFOA | 0.38 J ng/l | Laboratory contamination (see method blank qualifications). |
| | EtFOSAA | 1.2 J ng/l | The associated sample result (VAPJ-GW-21-25-01-100720) was nondetect. |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2015380-03 | 10/13/2020 8:53 | PFOA | 0.54 J ng/l | VAPJ-GW-21-25-01-100720 (K2009120-019)<br>VAPJ-GW-60-62-01-100720 (K2009120-023)<br>EQ-PAIL-01-100720 (K2009120-027)<br>EQ-AUGUR-01-100720 (K2009120-031)<br>EQ-TUBE-01-100720 (K2009120-032)<br>TRIP BLANK (K2009120-035) |
| KQ2015282-04 | 10/12/20 8:30 | EtFOSAA | 1.1 J ng/l | Associated results were nondetect. No qualifiers. |
| | | PFBA | 0.42 J ng/l | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | PFOA | 0.52 J ng/l | |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2016535-02 | 10/23/20 06:03 | PFOS | 0.57 J ng/L | No associated results. |
| | | PFOA | 0.62 J ng/L | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | EtFOSAA | 1.2 J ng/L | Associated results were nondetect. No qualifiers. |
| KQ2016536-04 (water) | 10/23/20 14:57 | PFOA | 0.59 J ng/L | VAPJ-GW-28-32-01-100720 (K2009120-021)<br>VAPJ-GW-60-62-01-100720 (K2009120-023)<br>EQ-PAIL-01-100720 (K2009120-027)<br>EQ-AUGUR-01-100720 (K2009120-031) |
| | | MeFOSA | 0.47 J ng/L | Associated results were nondetect. No qualifiers. |
| KQ2016536-04 (soil) | 10/23/20 14:57 | PFOA | 0.15 J ng/g | VAPE-SOIL-COMP-1-5-01-100520 (K2009120-002)<br>VAPE-SOIL-COMP-25-29-01-100520 (K2009120-007)<br>VAPE-SOIL-COMP-25-29-02-100520 (K2009120-008)<br>VAPE-SOIL-COMP-25-29-03-100520 (K2009120-009)<br>VAPF-SOIL-COMP-26-30-01-100620 (K2009120-012)<br>VAPF-SOIL-COMP-26-30-02-100620 (K2009120-013)<br>VAPF-SOIL-COMP-40-45-01-100620 (K2009120-015)<br>VAPF-SOIL-60-62-01-100620 (K2009120-016)<br>VAPF-SOIL-COMP-66-70-01-100620 (K2009120-018)<br>VAPJ-SOIL-COMP-21-25-01-100720 (K2009120-020)<br>VAPJ-SOIL-60-62-01-100720 (K2009120-024)<br>VAPJ-SOIL-COMP-70-74-01-100720 (K2009120-025)<br>VAPE-SOIL-COMP-36-40-01-100820 (K2009120-033) |
| | | MeFOSA | 0.12 J ng/g | VAPE-SOIL-COMP-25-29-01-100520 (K2009120-007)<br>VAPE-SOIL-COMP-25-29-03-100520 (K2009120-009)<br>VAPF-SOIL-COMP-40-45-01-100620 (K2009120-015)<br>VAPJ-SOIL-COMP-21-25-01-100720 (K2009120-020)<br>VAPJ-SOIL-60-62-01-100720 (K2009120-024)<br>VAPE-SOIL-COMP-36-40-01-100820 (K2009120-033) |
| KQ2016537-02 | 10/26/20 18:03 | PFOA | 0.51 J ng/L | EQ-TUBE-01-100720 (K2009120-032)<br>TRIP BLANK (K2009120-035) |
| | | MeFOSA | 0.52 J ng/L | Associated results were nondetect. No qualifiers. |
| KQ2016817-02 | 10/29/20 10:46 | PFOA | 0.50 J ng/L | Associated results were greater than 10 times the blank concentration. No qualifiers. |
| | | EtFOSA | 0.29 J ng/L | Associated results were nondetect. No qualifiers. |
| | | EtFOSAA | 1.3 J ng/L | |

**Labeled Analogs - surrogates (Section 4Biv):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists. Seventeen results were rejected (R) due to severely low labeled analog recoveries.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-6:2 FTS | 32 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-8:2 FTS | 22 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-PFDA | 13 | R |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-PFDoDA | 6 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-PFHxA | 24 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-PFTeDA | 4 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C2-PFUnDA | 10 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C3-HFPO-DA | 19 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C3-PFBS | 17 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFBA | 33 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFHpA | 20 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFOA | 19 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFOA | 13 | R: 11Cl-PF3OUdS 9Cl-PF3ONS ADONA |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFOS | 9 | |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C4-PFOS | 12 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C5-PFNA | 18 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 13C5-PFPeA | 31 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | 18O2-PFHxS | 18 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D3-MeFOSA | 23 | J+ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D3-MeFOSAA | 6 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D5-EtFOSA | 18 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D5-EtFOSAA | 8 | R |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D7-MeFOSE | 34 | UJ |
| VAPE-GW-1-5-01-100520 | K2009120-001 | D9-EtFOSE | 16 | R |
| VAPE-GW-16-20-01-100520 | K2009120-003 | 13C2-PFTeDA | 46 | UJ |
| VAPE-GW-25-29-01-100520 | K2009120-004 | 13C2-PFTeDA | 47 | UJ |
| VAPE-GW-25-29-02-100520 | K2009120-005 | 13C2-PFTeDA | 37 | UJ |
| VAPF-SOIL-COMP-26-30-02-100620 | K2009120-013 | 13C2-6:2 FTS | 47 | UJ |
| VAPF-SOIL-COMP-26-30-02-100620 | K2009120-013 | 13C2-PFHxA | 48 | UJ |
| VAPF-SOIL-COMP-26-30-02-100620 | K2009120-013 | 13C4-PFBA | 47 | UJ |
| VAPF-SOIL-COMP-40-45-01-100620 | K2009120-015 | 13C4-PFOA | 49 | J+ |
| VAPF-SOIL-60-62-01-100620 | K2009120-016 | 13C4-PFOA | 45 | J+ |
| VAPJ-GW-70-74-01-100720 | K2009120-026 | 13C2-PFTeDA | 47 | UJ |
| VAPJ-GW-70-74-01-100720 | K2009120-026 | 13C4-PFOA | 45 | J+ |
| EQ-PAIL-01-100720 | K2009120-027 | 18O2-PFHxS | 48 | UJ |
| RC3MAN-WAT-BULK-01-100820 | K2009120-028 | 13C2-PFTeDA | 43 | UJ |
| RC3MAN-WAT-BULK-01-100820 | K2009120-028 | 13C3-HFPO-DA | 48 | UJ |
| RC3MAN-WAT-BULK-01-100820 | K2009120-028 | 13C4-PFHpA | 46 | J+ |
| RC3MAN-WAT-BULK-01-100820 | K2009120-028 | D3-MeFOSAA | 42 | UJ |
| RC3MAN-WAT-BULK-01-100820 | K2009120-028 | D5-EtFOSAA | 40 | J+ |

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| OD2-WAT-BULK-01-100820 | K2009120-029 | 13C2-PFTeDA | 34 | UJ |
| OD2-WAT-BULK-01-100820 | K2009120-029 | D3-MeFOSAA | 43 | UJ |
| RC3CUL-WAT-BULK-01-100820 | K2009120-030 | 13C2-PFHxA | 43 | J+ |
| RC3CUL-WAT-BULK-01-100820 | K2009120-030 | 13C2-PFTeDA | 42 | UJ |
| RC3CUL-WAT-BULK-01-100820 | K2009120-030 | 13C3-HFPO-DA | 48 | UJ |
| RC3CUL-WAT-BULK-01-100820 | K2009120-030 | D3-MeFOSAA | 47 | UJ |
| VAPE-GW-36-40-01-100820 | K2009120-034 | 13C2-PFTeDA | 42 | UJ |
| VAPE-GW-36-40-01-100820 | K2009120-034 | 13C3-HFPO-DA | 46 | UJ |
| VAPE-GW-36-40-01-100820 | K2009120-034 | 13C4-PFHpA | 49 | J+ |
| VAPE-GW-36-40-01-100820 | K2009120-034 | D3-MeFOSAA | 46 | UJ |
| VAPE-GW-36-40-01-100820 | K2009120-034 | D5-EtFOSAA | 49 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (matrix) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2015282-03 (waters) | PFHpS | **146/**-- | 70-130 | J+:<br>VAPE-GW-1-5-01-100520 (K2009120-001)<br>VAPE-GW-16-20-01-100520 (K2009120-003)<br>VAPE-GW-25-29-01-100520 (K2009120-004)<br>VAPE-GW-25-29-02-100520 (K2009120-005)<br>VAPE-GW-25-29-03-100520 (K2009120-006) |
| | PFHxS | **132/**-- | 70-130 | |
| KQ2015380-01/ KQ2015380-02 (waters) | 11Cl-PF3OUdS | **133/136** | 70-130 | Associated results were nondetect. No qualifiers. |
| | 4:2 FTS | 120/**132** | 70-130 | Associated results were nondetect. No qualifiers. |
| | PFTeDA | **135/140** | 70-130 | |
| | PFTrDA | 115/**140** | 70-130 | |
| | NEtFOSE | **146/141** | 70-130 | J+:<br>VAPJ-GW-60-62-01-100720 (K2009120-023) |
| | PFHxS | 115/**136** | 70-130 | J+:<br>VAPF-GW-26-30-01-100620 (K2009120-010)<br>VAPF-GW-26-30-02-100620 (K2009120-011)<br>VAPF-GW-36-39-01-100620 (K2009120-014)<br>VAPF-GW-66-70-01-100620 (K2009120-017)<br>VAPJ-GW-21-25-01-100720 (K2009120-019)<br>VAPJ-GW-28-32-01-100720 (K2009120-021)<br>VAPJ-GW-70-74-01-100720 (K2009120-026)<br>RC3MAN-WAT-BULK-01-100820 (K2009120-028)<br>OD2-WAT-BULK-01-100820 (K2009120-029)<br>RC3CUL-WAT-BULK-01-100820 (K2009120-030)<br>VAPE-GW-36-40-01-100820 (K2009120-034) |

**Matrix spike/matrix spike sample duplicate (Section 6Av):** Detects associated with MS/MSD exceedances were qualified as summarized below:

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **VAPE-GW-25-29-03-100520:** | | | |
| PFPeS | **63/**86 | 70-130 | The results for sample VAPE-GW-25-29-01-100520, VAPE-GW-25-29-02-100520 and VAPE-GW-25-29-01-100520 and VAPE-GW-25-29-03-100520 were qualified as estimated biased low (J-). |
| PFHxS | **143/**120 | 70-130 | The detects for sample VAPE-GW-25-29-01-100520, VAPE-GW-25-29-02-100520 and VAPE-GW-25-29-01-100520 and VAPE-GW-25-29-03-100520 were qualified as estimated biased high (J+). |
| PFHpS | **162/159** | 70-130 | |
| PFNS | **131/**123 | 70-130 | |
| PFHxA | **131/147** | 70-130 | |
| 11Cl-PF3OUdS | **145/146** | 70-130 | The results for sample VAPE-GW-25-29-01-100520, VAPE-GW-25-29-02-100520, and VAPE-GW-25-29-01-100520 and VAPE-GW-25-29-03-100520 were nondetect. No qualifiers. |
| PFTrDA | **145/**123 | 70-130 | |
| PFOS | **322/247** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFBA | **-59/-34** | 70-130 | |
| PFOA | **241/287** | 70-130 | |

Any R qualifiers take precedence over other qualifiers. Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2009515 Report date (mm/dd/yyyy): 11/6/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/30/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | A trip blank was recieved; however, was not listed on the COC. |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | | ☐ | ☒ | ☐ | MN limits used for review. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Do any of the analyses contain samples for field or trip blanks? | | ☒ | ☐ | ☐ | One trip blank reported and two equipment blanks (EQ) reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **B.** | Do method blanks for any analyses contain target analytes above the reporting limit? | | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | Do instrument blanks contain analytes above the reporting limit? | | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are the lab recovery limits for surrogates specified on the report? | | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☒ | ☐ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>soil/sed limits used: 50% to 150% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSDs: MW12A-GW-256-260-03-101520 ( K2009515-013)<br>MW12A-SOIL-COMP-226-230-03-101420 (K2009515-009) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs, MSDs<br>FDs: MW12A-SOIL-COMP-226-230-01-101420/MW12A-SOIL-COMP-226-230-02-101420/MW12A-SOIL-COMP-226-230-03-101420<br>MW12A-GW-256-260-01-101520/MW12A-GW-256-260-02-101520/MW12A-GW-256-260-03-101520 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water) or ±RL<br>50% limit for PFAS (Soil/Sed) or ±RL<br>LCSD, MSDs and FDs - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/30/2020

## Data Validation Qualifications for K2009515

**Trip blank and equipment blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified B.

| Blank ID | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|
| TRIP BLANK | PFOA | 0.50 J ng/l | Laboratory contamination (see method blank qualifications). |
| | MeFOSA | 0.50 J ng/l | MW12A-SOIL-48.5-49.5-01-101220 (K2009515-002)<br>MW12A-GW-96-100-01-101320 (K2009515-004)<br>MW12A-SOIL-COMP-226-230-02-101420 (K2009515-008)<br>MW12A-GW-256-260-02-101520 (K2009515-012)<br>MW12A-SOIL284-288-01-101520 (K2009515-014)<br>0D1-WAT-BULK-01-101320 (K2009515-017) |
| EQ-DRILLWAT-01-101220 | PFOA | 0.69 J ng/l | Laboratory contamination (see method blank qualifications). |
| | FOSA | 0.67 J ng/l | Results used to check drilling water. No qualifiers. |
| EQ-DAKOTAPUMP-01-101620 | PFOA | 0.41 J ng/l | Laboratory contamination (see method blank qualifications). |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2016452-04 | 10/21/2020 15:55 | MeFOSA | 0.17 J ng/l | MW12A-SOIL-48.5-49.5-01-101220 (K2009515-002)<br>MW12A-SOIL-COMP-226-230-02-101420 (K2009515-008)<br>MW12A-SOIL284-288-01-101520 (K2009515-014) |
| KQ2016181-04 | 10/22/2020 8:51 | PFOA | 0.53 J ng/l | EQ-DRILLWAT-01-101220 (K2009515-003)<br>EQ-DAKOTAPUMP-01-101620 (K2009515-019)<br>TRIP BLANK (K2009515-020) |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2016588-02 | 10/27/20 10:59 | MeFOSA | 0.10 ng/g | MW12A-SOIL-48.5-49.5-01-101220 (K2009515-002)<br>MW12A-SOIL-COMP-226-230-02-101420 (K2009515-008)<br>MW12A-SOIL284-288-01-101520 (K2009515-014) |
| KQ2016589-02 | 10/27/20 22:57 | PFOA | 0.43 J ng/L | EQ-DRILLWAT-01-101220 (K2009515-003)<br>EQ-DAKOTAPUMP-01-101620 (K2009515-019)<br>TRIP BLANK (K2009515-020) |

**Labeled Analogs - surrogates (Section 4Biv):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW12A-GW-176-180-01-01320 | K2009515-005 | 13C3-HFPO-DA | 49 | UJ |
| MW12A-GW-256-260-03-101520 | K2009515-013 | 13C2-PFHxA | 47 | J+ |
| RC3MAN-WAT-BULK-01-101320 | K2009515-015 | 13C2-PFHxA | 45 | J+ |
| RC3MAN-WAT-BULK-01-101320 | K2009515-015 | 13C2-PFTeDA | 38 | UJ |
| RC3MAN-WAT-BULK-01-101320 | K2009515-015 | 13C3-HFPO-DA | 42 | UJ |
| RC3MAN-WAT-BULK-01-101320 | K2009515-015 | D5-EtFOSA | 48 | UJ |
| RC3CUL-WAT-BULK-01-101320 | K2009515-016 | 13C2-4:2 FTS | 208 | No Qualifier |
| RC3CUL-WAT-BULK-01-101320 | K2009515-016 | 13C2-PFHxA | 48 | J+ |
| RC3CUL-WAT-BULK-01-101320 | K2009515-016 | 13C3-HFPO-DA | 48 | UJ |
| RC3CUL-WAT-BULK-01-101320 | K2009515-016 | D3-MeFOSAA | 49 | UJ |
| 0D1-WAT-BULK-01-101320 | K2009515-017 | 13C2-PFTeDA | 40 | UJ |
| 0D2-WAT-BULK-01-101320 | K2009515-018 | 13C2-PFHxA | 46 | J+ |
| 0D2-WAT-BULK-01-101320 | K2009515-018 | 13C2-PFTeDA | 35 | UJ |

**Matrix spike/matrix spike sample duplicate (Section 6Av):** MS/MSD exceedances are summarized below. Results did not require qualification.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **MW12A-SOIL-COMP-226-230-03-101420:** | | | |
| PFPeS | 146/**154** | 50-150 | The result for samples MW12A-SOIL-COMP-226-230-01-101420, MW12A-SOIL-COMP-226-230-02-101420 and MW12A-SOIL-COMP-226-230-03-101420 were nondetect. No qualifiers. |
| **MW12A-GW-256-260-03-101520:** | | | |
| PFTeDA | 109/**131** | 70-130 | The results for samples MW12A-GW-256-260-01-101520 , MW12A-GW-256-260-02-101520, and MW12A-GW-256-260-03-101520 were nondetect. No qualifiers. |
| 6:2 FTS | 107/**133** | 70-130 | |
| PFBA | 93/**58** | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |

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Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2009831 Report date (mm/dd/yyyy): 11/11/2020

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 11/30/2020

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | The sample receipt form indicated a trip blank was recieved; however, was not listed on the COC. This sample was logged in as a Field Blank.<br>One of the IDs on the sample label did not match the COC. The laboratory was able to identify the sample by the process of elimination. |
| **C.** | Were there samples preserved according to program requirements? | | ☐ | ☒ | ☐ | Sample receipt temperature is 10.9 °C. See data qualification summary for qualifications. |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |

| | | | | | |
|---|---|---|---|---|---|
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for summary of exceedances and qualifications. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | One field blank reported. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☒ | ☐ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☒ | ☐ | |
| | ii. | Below the lab QC limits? | ☒ | ☐ | ☐ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☒ | ☐ | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☒ | ☐ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>soil/sed limits used: 50% to 150% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☒ | ☐ | ☐ | See data qualification summary for summary of qualifications. |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☒ | ☐ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSDs: MW12A-GW-256-260-03-101520 (K2009515-013)<br>MW12A-SOIL-COMP-226-230-03-101420 (K2009515-009) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☐ | ☒ | ☐ | |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☒ | ☐ | ☐ | See data qualification summary for summary of exceedances and qualifications. |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☒ | ☐ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSDs, MSDs<br>FDs: MW2D-GW-7-17-01-102120/MW2D-GW-7-17-02-102120<br>MW2C-GW-35-40-01-102120/ MW2C-GW-35- |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | 40-02-102120/ MW2C-GW-35-40-03-102120<br>MW13D-GW-20-30-01-102120/MW13D-GW-20-30-02-102120 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | ☒ | ☐ | ☐ | 30% limit for PFAS (water) or ±RL<br>50% limit for PFAS (Soil/Sed) or ±RL<br>LCSD, MSD and FD - ok |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualification.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 11/30/2020

## Data Validation Qualifications for K2009831

**Chain of custody preservation, and holding time (Section 1C):** Samples were received at 10.9 °C and results were qualified (UJ and J-) due to the sample receipt temperature.

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV KC2000572 (11/03/20 18:50) | NEtFOSE | 145 | 70-130% | NEtFOSE results not reported from this CCV. |
| CCV KC2000572 (11/04/20 12:59) | NEtFOSE | 274 | 70-130% | J+: MW15A-GW-296-300-01-102620 (K2009831-010) |
| | NMeFOSE | 155 | 70-130% | Associated results were nondetect. No qualifiers. |
| | EtFOSAA | 65.5 | 70-130% | EtFOSAA results not reported from this CCV. |

**Field blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified B.

| Blank ID | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|
| FIELD BLANK | PFOA | 0.40 J ng/l | Laboratory contamination (see method blank qualifications). |
| | NEtFOSE | 0.13 J ng/l | MW9A-GW-140-150-01-102300 (K2009831-008)<br>MW15A-GW-296-300-01-102620 (K2009831-010)<br>MW3A-GW-230-250-01-102120 (K2009831-012)<br>MW6B-GW-140-150-01-102120 (K2009831-018)<br>MW6A-GW-185-192-01-102120 (K2009831-019) |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2016846-04 | 10/30/2020 7:49 | PFOA | 0.52 J ng/l | MW13A-GW-350-370-01-102320 (K2009831-007)<br>MW12A-GW-350-360-01-102620 (K2009831-009)<br>MW6B-GW-140-150-01-102120 (K2009831-018)<br>FIELD BLANK (K2009831-023) |
| KQ2017040-03 | 11/2/2020 8:39 | PFOA | 0.42 J ng/l | MW15A-GW-296-300-01-102620 (K2009831-010) |
| | | MeFOSA | 0.64 J ng/l | Associated results were nondetect. |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2016991-02 (water) | 10/30/20 14:53 | PFOA | 0.45 J ng/L | Associated results were nondetect. |
| KQ2017100-04 (waters) | 11/2/20 15:58 | EtFOSAA | 1.2 J ng/L | MW3B-GW-110-130-01-102120 (K2009831-013) |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2017233-04 (waters) | 11/4/20 13:10 | MeFOSA | 0.84 J ng/L | Associated results were nondetect. |

**Labeled Analogs - surrogates (Section 4Biv):** Results associated with low recoveries were qualified as estimated (UJ and J+), as these labeled analogs are used to quantitate the analyte of interest and an inverse relationship exists.

| Sample ID | Lab ID | Labeled Analog | % Recovery (Limits 50% to 200%) | Qualification of Associated non-Labeled Analyte |
|---|---|---|---|---|
| MW13A-GW-350-370-01-102320 | K2009831-007 | 13C2-PFHxA | 49 | UJ |
| MW13A-GW-350-370-01-102320 | K2009831-007 | 13C3-HFPO-DA | 43 | UJ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | 13C2-PFTeDA | 49 | UJ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | 13C4-PFOA | 49 | J+ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | D3-MeFOSA | 42 | UJ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | D5-EtFOSA | 45 | UJ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | D7-MeFOSE | 44 | UJ |
| MW15A-GW-296-300-01-102620 | K2009831-010 | D9-EtFOSE | 33 | J+ |
| MW3A-GW-230-250-01-102120 | K2009831-012 | 13C2-PFDoDA | 47 | UJ |
| MW3A-GW-230-250-01-102120 | K2009831-012 | D3-MeFOSAA | 33 | UJ |
| MW3A-GW-230-250-01-102120 | K2009831-012 | D5-EtFOSAA | 32 | UJ |
| MW3B-GW-110-130-01-102120 | K2009831-013 | D3-MeFOSAA | 48 | UJ |
| MW5A-GW-210-220-01-102120 | K2009831-014 | 13C2-PFHxA | 48 | UJ |
| MW5A-GW-210-220-01-102120 | K2009831-014 | 13C3-HFPO-DA | 48 | UJ |
| MW5A-GW-210-220-01-102120 | K2009831-014 | 13C8-FOSA | 46 | UJ |
| MW5A-GW-210-220-01-102120 | K2009831-014 | D3-MeFOSAA | 46 | UJ |

**Laboratory control sample/Laboratory control sample duplicate (Section 5Bi):** Detects associated with high recoveries were qualified as J+:

| LCS/LCSD IDs (matrix) | Compound | LCS/LCSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|---|
| KQ2017040-01/ KQ2017040-02 (waters) | EtFOSE | **236/255** | 70-130 | J+:<br>MW9A-GW-140-150-01-102300 (K2009831-008)<br>MW3A-GW-230-250-01-102120 (K2009831-012)<br>MW6B-GW-140-150-01-102120 (K2009831-018)<br>MW6A-GW-185-192-01-102120 (K2009831-019)<br>FIELD BLANK (K2009831-023) |
| | MeFOSE | **138/**122 | 70-130 | Associated results were nondetect. No qualifiers. |
| KQ2016846-03 | 4:2 FTS | **132/**-- | 70-130 | Associated results were nondetect. No qualifiers. |
| | 6:2 FTS | **131/**-- | 70-130 | |
| | EtFOSAA | **142/**-- | 70-130 | |
| | PFPeS | **131/**-- | 70-130 | |

**Matrix spike/matrix spike sample duplicate (Section 6Av):** MS/MSD exceedances and results qualified are summarized below.

| Compound | MS/MSD % Recovery | Recovery Limits | Qualifiers |
|---|---|---|---|
| **MW2C-GW-35-40-03-102120:** | | | |
| 4:2 FTS | **128/133** | 70-130 | The results for samples MW2C-GW-35-40-01-102120, MW2C-GW-35-40-02-102120, and MW2C-GW-35-40-03-102120 were nondetect. No qualifiers. |
| 6:2 FTS | 111/**137** | 70-130 | |
| 10:2 FTS | 114/**133** | 70-130 | |
| EtFOSAA | 106/**135** | 70-130 | |
| PFDS | **131/139** | 70-130 | |
| PFBA | **150/142** | 70-130 | The detects for samples MW2C-GW-35-40-01-102120, MW2C-GW-35-40-02-102120, and MW2C-GW-35-40-03-102120 were qualified estimated biased high (J+) |
| PFNS | **132/144** | 70-130 | |
| PFOA | **155**/120 | 70-130 | The sample concentration was greater than 4 times the spike concentration. No qualifiers. |
| PFOS | **648/643** | 70-130 | |

Results with both high bias (J+) and low bias (J-), should be qualified as estimated (J).

MINNESOTA

Minnesota Department of Agriculture 651-201-6148
Minnesota Department of Health 651-201-5357
Minnesota Pollution Control Agency 651-757-2560

# Laboratory data review checklist

*Doc Type: Data Review*

**Instructions:** The following is the informal checklist that should be used to review data for the Minnesota Department of Agriculture, Minnesota Pollution Control Agency, and Minnesota Department of Health. The information follows the general format of the National Functional Guidelines, which is the primary data review tool used in the U.S. Environmental Protection Agency's Contract Laboratory Program for Superfund analytical work. Refer to the appropriate guidance document for each agency for instructions.

## Project information

Project name: Project 1007

Work order number/Lab report ID: K2011896 Report date (mm/dd/yyyy): 1/13/2021

Laboratory: ALS - Kelso, Washington Review date (mm/dd/yyyy): 1/19/2021

## 1. Chain of custody, preservation, and holding times

| Questions | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Is there a chain of custody (COC) with the report? | | ☒ | ☐ | ☐ | |
| **B.** | Is there a sample condition form with the report? | | ☒ | ☐ | ☐ | |
| **C.** | Were there samples preserved according to program requirements? | | ☒ | ☐ | ☐ | |
| **D.** | Were samples received in the correct containers? | | ☒ | ☐ | ☐ | |
| | i. | Was there enough sample volume/weight to complete all requested analyses? | ☒ | ☐ | ☐ | |
| | ii. | Was there enough sample collected to complete required batch QC? | ☒ | ☐ | ☐ | |
| **E.** | Were samples received within holding time for sample prep for all requested analyses? | | ☒ | ☐ | ☐ | |
| **F.** | Are there notes about sample condition or holding time issues on the COC? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **G.** | Are there narration or data qualifiers with the report about sample condition or holding time issues? Explain the data impact. | | ☐ | ☒ | ☐ | |
| **H.** | Are lab IDs cross-referenced correctly with the field IDs. | | ☒ | ☐ | ☐ | Yes, but no sample summary table included in the report. |

## 2. Calibration

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Do the report narrative or data qualifiers indicate calibration problems for any analyses? If yes, explain the data impact. | ☒ | ☐ | ☐ | MN limits used for review.<br>See data qualification summary for summary of exceedances. No qualifications required. |

## 3. Blanks

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Do any of the analyses contain samples for field or trip blanks? | ☒ | ☐ | ☐ | A trip blank was included. |
| | i. | If yes, are there target analytes present above the reporting limit in the blanks? | ☐ | ☒ | ☐ | Detects were less than the RL.<br>Results for PFESAs and PFECAs were not reported for the trip blank. |
| | ii. | If yes, are the same compounds also present in the samples? Explain possible data impact. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **B.** | | Do method blanks for any analyses contain target analytes above the reporting limit? | ☐ | ☒ | ☐ | Blank detects were below the RL. |
| | i. | If yes, are the same compounds present in the samples? | ☒ | ☐ | ☐ | |
| | ii. | Is the amount of target analyte in the method blank more than 1/10$^{th}$ of that in the sample(s)? Explain the possible impact on sample results. | ☒ | ☐ | ☐ | See data qualification summary for summary of detects and qualifications. |
| **C.** | | Do instrument blanks contain analytes above the reporting limit? | ☐ | ☒ | ☐ | Detects were below the RL. See data qualification summary for summary of detects and qualifications. |

## 4. Surrogates or organic analysis

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | | Are the lab recovery limits for surrogates specified on the report? | ☒ | ☐ | ☐ | MPCA limits used: 50% to 200% |
| **B.** | | Are the surrogates outside lab QC limits? (These should have a data qualifier.) | ☐ | ☒ | ☐ | |
| | i. | If yes, are the surrogates above the lab QC limits? | ☐ | ☐ | ☒ | |
| | ii. | Below the lab QC limits? | ☐ | ☐ | ☒ | |
| | iii. | Were the affected samples re-analyzed? Discuss in the case narrative. | ☐ | ☐ | ☒ | |
| | iv. | Explain what this could mean for the affected samples. Include in narrative. | ☐ | ☐ | ☒ | |

## 5. Laboratory control sample/Laboratory control sample duplicate (LCS/LCSD)

| Question | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|
| **A.** | Are there LCS/LCSD samples present for the reporting analyses? | | ☒ | ☐ | ☐ | |
| **B.** | Are there LCS/LCSD compounds outside lab limits? If the LCS/LCSD fails, the LCS/LCSD and samples must be re-analyzed. | | ☐ | ☒ | ☐ | MPCA PFAS water limits used: 70% to 130%.<br>soil/sed limits used: 50% to 150% |
| | i. | If yes, are there compounds above the lab QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |
| | ii. | Below the QC limits? If yes, an explanation is required. Include in narrative. | ☐ | ☐ | ☒ | |

## 6. Matrix spike/Matrix spike duplicate/Sample duplicate (MS/MSD/DUP)

| Question | | | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|---|---|
| **A.** | Do the analytical methods used require an MS and/or MSD? If no, skip to 6.B. | | | ☒ | ☐ | ☐ | |
| | i. | Have the required matrix spikes been prepared and reported? | | ☒ | ☐ | ☐ | MS/MSDs: MW14B-SOIL-COMP-30-37-03-121520 (K2011896-004) |
| | ii. | If no, is there and explanation in the report as to why? | | ☐ | ☐ | ☒ | |
| | iii. | Did the lab process an alternate spiked sample (such as LCSD) instead? | | ☒ | ☐ | ☐ | LCSD |
| | iv. | Are the lab QC limits specified on the report? | | ☒ | ☐ | ☐ | |
| | v. | Are there compounds outside the lab QC limits? | | ☐ | ☒ | ☐ | |
| | vi. | If yes, did the lab re-run an MS/MSD? | | ☐ | ☐ | ☒ | |
| | | 1. | Did the re-run MS/MSD pass? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 2. | Did the re-run MS/MSD fail? Discuss the case narrative. | ☐ | ☐ | ☒ | |
| | | 3. | Is the source sample also flagged for MS/MSD compounds outside the lab QC limits? | ☐ | ☐ | ☒ | |
| **B.** | Was a duplicate sample submitted for the analytical method(s)? | | | ☒ | ☐ | ☐ | LCSD, MSD<br>FDs: MW14B-SOIL-COMP-30-37-02-121520/MW14B-SOIL-COMP-30-37-03-121520 |
| | i. | Is the Relative Percentage Difference (RPD) within 20%* for the duplicate pair?<br>If no, explain possible causes and data impact.<br>**Other RPDs may be acceptable. Check with regulatory agency.* | | ☐ | ☒ | ☐ | 30% limit for PFAS (water) or ±RL<br>50% limit for PFAS (Soil/Sed) or ±RL<br>LCSD, MSD - ok<br>FD: See data qualification summary for field duplicate qualifications. |

## 7. Method detection limits/Report limits

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are reporting limits clearly listed on the report for all analyses? | ☒ | ☐ | ☐ | |
| **B.** | Do the reporting limits meet the program required limits listed? If not, an explanation is required. | ☐ | ☒ | ☐ | Results for 11Cl-PF3OUdS, 9Cl-PF3ONS and ADONA were nondetect and reported to the RL, while other PFAS compound were reported to the method detection limit (MDL). |

## 8. Sample information

| Questions | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Are sample numbers cross-referenced correctly with the associated QC? | ☒ | ☐ | ☐ | |
| **B.** | Are soil samples reported in dry weight basis? | ☒ | ☐ | ☐ | |
| **C.** | Are percent moisture results reported? | ☒ | ☐ | ☐ | |
| **D.** | Are positive detections reported? | ☒ | ☐ | ☐ | |
| **E.** | Are sample analytes appropriately flagged if the QC failed? | ☒ | ☐ | ☐ | |

## 9. Report narrative

| Question | | Yes | No | N/A | Comments |
|---|---|---|---|---|---|
| **A.** | Is a narrative provided with the laboratory report which describes all problems with the analyses and all corrective actions taken to address these problems? | ☒ | ☐ | ☐ | |

## 10. Additional comments about the lab report

**Reviewed using MPCA Guidance on Perfluorochemicals Analysis (July 2020).**

**See attachedment for qualifications.**

## Certification

*By typing my name below, I certify the above statements to be true and correct, to the best of my knowledge, and that this information can be used for the purpose of processing this form.*

**Authorized Representative**

Name: Lisa Smith Title: Environmental Chemist (CEAC)

*(This document has been electronically signed.)* Date (mm/dd/yyyy): 1/19/2020

## Data Validation Qualifications for K2011896

**Calibration (Section 2):** Calibration noncompliances and the results qualified are summarized below.

| Calibration (Date) | Analyte | %R | Limit | Qualifiers |
|---|---|---|---|---|
| CCV K2011896 (01/06/20 09:42) | 13C2-6:2 FTS | 233 | 50-200% | Only PFBA, PFHxS and PFOA reported from this CCV. No qualifiers. |
| | 13C2-8:2 FTS | 217 | 50-200% | |

**Trip blank detections (Section 3Aii):** Detects within 10 times the blank concentrations were qualified B.

| Blank ID | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|
| Trip Blank | 6:2 FTS | 0.71 J ng/l | MW14B-GW-29-37-01-121520 (K2011896-005) |
| | PFOS | 0.77 J ng/l | Laboratory contamination (see method blank qualifications). |

**Method blank and instrument blank detections (Section 3Bii and C):** Detects within 10 times the blank concentrations were qualified B.

| Method Blank ID | Prep Date | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2020376-03 | 12/18/2020 | PFOS | 0.47 J ng/l | Trip Blank (K2011896-007) |
| KQ2020545-04 | 12/21/2020 | PFPeA | 0.28 J ng/g | MW14B-SOIL-COMP-10-30-01-121520 (K2011896-001)<br>MW14B-SOIL-COMP-30-37-01-121520 (K2011896-002)<br>MW14B-SOIL-COMP-30-37-02-121520 (K2011896-003)<br>MW14B-SOIL-COMP-30-37-03-121520 (K2011896-004)<br>MW14B-SOIL-COMP-56-60-01-121520 (K2011896-006) |
| | | MeFOSA | 0.14 J ng/g | MW14B-SOIL-COMP-10-30-01-121520 (K2011896-001)<br>MW14B-SOIL-COMP-30-37-02-121520 (K2011896-003)<br>MW14B-SOIL-COMP-30-37-03-121520 (K2011896-004)<br>MW14B-SOIL-COMP-56-60-01-121520 (K2011896-006) |

| CCB Blank ID | Date/Time | Compound | Blank Concentration | Results Qualified B |
|---|---|---|---|---|
| KQ2020758-02 (soil) | 1/1/21 04:35 | PFPeA | 0.29 J ng/g | MW14B-SOIL-COMP-10-30-01-121520 (K2011896-001)<br>MW14B-SOIL-COMP-30-37-01-121520 (K2011896-002)<br>MW14B-SOIL-COMP-30-37-02-121520 (K2011896-003)<br>MW14B-SOIL-COMP-30-37-03-121520 (K2011896-004)<br>MW14B-SOIL-COMP-56-60-01-121520 (K2011896-006) |
| | | MeFOSA | 0.12 J ng/g | MW14B-SOIL-COMP-10-30-01-121520 (K2011896-001)<br>MW14B-SOIL-COMP-30-37-02-121520 (K2011896-003)<br>MW14B-SOIL-COMP-30-37-03-121520 (K2011896-004)<br>MW14B-SOIL-COMP-56-60-01-121520 (K2011896-006) |
| KQ2020758-04 (waters) | 1/1/21 04:35 | MeFOSA | 0.46 J ng/L | Associated results were nondetect. |
| 1/6/21 09:53 (soil) | 1/6/21 09:53 | PFPeA | 0.34 J ng/g | PFPeA not reported from this run. |
| KQ2100372-03 (waters) | 1/8/21 17:05 | PFBA | 1.7 J ng/L | Only PFOS (waters) reported from this run. No qualifiers. |

**Field duplicate results (Section 6Bi):** Detects associated with high RPDs were qualified as estimated (J):

| Analyte | Parent Sample Result (ng/L) | Field Duplicate Result (ng/L) | Triplicate Result (ng/L) | RPD (%) |
|---|---|---|---|---|
| **MW14B-SOIL-COMP-30-37-01-121520/ MW14B-SOIL-COMP-30-37-02-121520/ MW14B-SOIL-COMP-30-37-03-121520:** | | | | |
| PFOS | 6.2 | 3.1 | 5.3 | 64 |