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PFAS Analytical Method Guidance: NPDES and Residuals Programs

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Overview

This document contains guidance for improving PFAS (Per and Polyfluoroalkyl Substances) testing of wastewater media and residuals using EPA Method 1633.

- **PFAS testing for wastewater influent, effluent, and sludge** is required by National Pollutant Discharge Elimination Systems (NPDES) and Surface Water Discharge (SWD) permits.
- **PFAS testing for residuals** (materials for land application) is required in the Approvals of Suitability (AOSs).

This document contains steps from EPA Method 1633 and specifies notes to make in the laboratory narrative. The document also contains guidance external to, but allowed by, EPA Method 1633 that will improve data quality and reliability.

Note that this guidance was compiled referencing the PFAS Compendium of Analytical Methods document from the Massachusetts Department of Environmental Protection (MassDEP) Bureau of Waste Site Cleanup.

Contact MassDEP.NPDES@mass.gov for any questions about this document. Include "PFAS Analytical Method Guidance" in the subject line.

Analytical method guidance

1. **Annual confirmation**
   By March 1st of each year, confirm whether the laboratory intends to follow this PFAS analytical method guidance in the current calendar year. List any anticipated challenges with following the guidance. Send annual confirmation by email to MassDEP.NPDES@mass.gov with subject line "Annual PFAS guidance confirmation, [lab name]."

2. **Laboratory Narrative**
   Include a narrative section in the lab report. List the details recommended in this guidance document, other quality control (QC) issues, and any method modifications.

3. **Reporting Limits (RLs)**
   Normal reporting limit ranges are listed in *Table 1. Reporting Limit (RL) ranges*.
   If reporting limits exceed the maximum level for any analyte listed in the table, explain in the laboratory narrative why lower reporting limits could not be achieved.
   Note that the sludge RL range is expressed in wet weight. In the .txt file submitted to eDEP, report dry weight concentrations for sludge samples (i.e. ng/g dry weight) per EPA Method 1633.

4. **Initial Precision and Recovery (IPR)**
   Per EPA Method 1633 each time a modification is made to the method, repeat the IPR procedure. Such modifications include alternative extraction procedures such as subsampling and centrifuging. Maintain records of any modifications made to the method, including the accompanying IPR, and make these available to MassDEP upon request.

5. **Subsampling of aqueous samples**
   Avoid subsampling aqueous samples (i.e. measured in ng/L) whenever possible.
   If subsampling is unavoidable, vortexing or shaking samples prior to subsampling could mitigate stratification or accumulation at air/water interface.
   If subsampling is used, explain in the laboratory narrative why an alternative method was not used and the method(s) used to homogenize the sample.
   If subsampling is consistently required for a given facility and matrix, coordinate with the facility to determine a more appropriate sample size.

6. **Extracted Internal Standards (EIS) equilibration**
   Equilibrate all sample matrices with the EIS for a minimum of 30 minutes.

7. **Test for total PFAS in aqueous samples**
   Test for total PFAS in aqueous samples (i.e. measured in ng/L), not just the soluble fraction. Include particulates in the analysis. Do not filter samples.
   If the sample is expected to clog the Solid Phase Extraction (SPE) cartridge, attempt method (a), (b), or (c) below. Select a method using best professional judgement. Options (a) and (b) are preferred to option (c). Subsampling may be performed if one these methods fail.
   Report any non-standard sample processing steps taken to manage difficult matrices in the laboratory narrative.
   Reference EPA Method 1633 for detailed directions for each step.

   a. **Settle and rinse particulates:** Add EIS to the entire sample, equilibrate for 30 minutes, put aqueous phase through Solid Phase Extraction (SPE), solvent (e.g. methanolic ammonium hydroxide) rinse the remaining particulates in the bottle, and add solvent rinse to SPE.

   b. **Centrifuge, one SPE:** Add EIS to the entire sample, equilibrate for 30 minutes, centrifuge, put aqueous phase through SPE, solvent (e.g. methanolic ammonium hydroxide) rinse the remaining particulates in the bottle after centrifuging, and add solvent rinse to SPE.

   c. **Centrifuge, separate SPE:** Add EIS to the entire sample, equilibrate for 30 minutes, centrifuge, separate solid and aqueous phases, extract aqueous and solid phases separately, and combine extracts prior to analysis.

8. **High PFAS samples**
   If a matrix is suspected to contain high PFAS concentrations, request a smaller sample size for future samples.

9. **Sludge processed as aqueous samples**
   Most NPDES sludge samples and AOS residuals samples can be processed as a solid (i.e. results in ng/g dry weight). If a sludge or residuals sample is processed as an aqueous sample (i.e. results in ng/L), justify in the laboratory narrative why this was chosen. Measure and report Total Solids (TS) for the sample using the TS procedure outlined in *Method 1633 11.1.2 Determination of percent solids in soils, sediments, and biosolids*. MassDEP recommends testing sludge above 2% TS as a solid whenever possible.

10. **Re-extraction**
    If re-extraction is performed on certain analytes due to QC failures, omit the original extraction results for those analytes in the .txt file submitted to eDEP. Instead,

include the re-extraction results for any re-extracted analytes and the original extraction results for the other analytes. The final .txt file will then contain the final results for each PFAS analyte. All analytes from the same sample will have the same sample ID.

11. **J qualified data**
    Report results down to the detection limit. This means that results above the detection limit but below the reporting limit should be uploaded with a qualifier (i.e. J-qualified values).

12. **QC recovery issues for non-detect results**
    In the .txt file submitted to eDEP, qualify any analytes with the following QC issues. Report the analyte result and use the qualifier "R" to indicate the uncertainty of the non-detect result. Make note of any R-qualified analytes in the laboratory narrative.

    a. **Ongoing Precision Recovery (OPR)**
       Recovery <10%; this affects non-detect results for the affected analyte in all samples extracted with this OPR.

    b. **Extracted Internal Standards (EIS)**
       Recovery <5% and S/N < 10:1; this affects associated non-detect results in the affected sample.

    c. **Non-Extracted Internal Standards (NIS)**
       Recovery <5% and S/N < 10:1; this affects associated non-detect results in the affected sample.
       Add the R qualifier in this instance based on professional judgement. If the percent recovery of the associated EIS is within the acceptance criteria, rejection may not be warranted. If the NIS percent rejection is low due to a bad injection or significant matrix suppression, adding an R qualifier to the associated non-detect results may be warranted.

    d. **Matrix Spike / Matrix Spike Duplicate (MS/MSD)**
       Recovery <10%; this affects non-detect results for the affected analyte in unspiked samples only.

13. **QC identification and quantitation criteria**
    In .txt file submitted to eDEP, qualify any positively identified analytes when the confirmation ion is not present. This is only applicable to target PFAS analytes with confirmation ions. Use the qualifier "NJ" to indicate that the analyte is "tentatively identified" or "presumptively" present. The NJ qualifier is not applicable for PFAS

without confirmation ions. Make note of any NJ-qualified analytes in the laboratory narrative.

Table 1. Reporting Limit (RL) ranges[1]

Target PFAS	Aqueous RL Range (ng/L)[2]	Sludge RL Range (ng/g, wet weight)[3]
PFBA	4 - 16	6.4 - 16
PFPeA	2 - 8	3.2 - 8
PFHxA, PFHpA, PFOA	1 - 4	1.6 - 4
PFNA	1 – 4	1.6 - 13
PFDA	1 – 4	1.6 - 4
PFUnA	1 – 4	1.6 - 5
PFDoA, PFTrDA, PFTeDA	1 – 4	1.6 - 4
PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoS	1 – 4	1.6 - 4
4:2 FTS, 6:2 FTS, 8:2 FTS	4 - 15	6.4 - 15
PFOSA, NMeFOSA, NEtFOSA, NMeFOSAA, NEtFOSAA	1 – 4	1.6 - 4
NMeFOSE, NEtFOSE	10 - 40	16 - 40
HFPO-DA, ADONA	2 – 8	6.4 - 16
PFMPA, PFMBA	4 - 16	3.2 - 8
NFDHA	2 – 7	3.2 - 8
9Cl-PF3ONS, 11Cl-PF3OUdS	4 - 15	6.4 - 15
PFEESA	2 – 8	3.2 - 7
3:3 FTCA	5 - 20	8 - 50
5:3 FTCA, 7:3 FTCA	25 - 100	40 – 100

[1] These Reporting Limit ranges are from EPA Method 1633A, Table 9.

[2] The aqueous RL ranges are applicable to wastewater treatment plant influent, effluent, and any sludge or residuals measured as an aqueous sample (i.e. in ng/L).

[3] The sludge RL ranges were taken from EPA Method 1633A Table 9, Solids column, multiplied by 10 per the footnote to account for the dry weight sample size.