



Department of Environmental Protection

100 Cambridge Street Suite 900 Boston, MA 02114 • 617-292-5500

Maura T. Healey
Governor

Kimberley Driscoll
Lieutenant Governor

Rebecca L. Tepper
Secretary

Bonnie Heiple
Commissioner

MassDEP-Required Laboratory Procedures for Testing PFAS in Residuals- Revised

From: MassDEP Residuals Program

Date: June 23, 2023

Summary of changes

MassDEP continues to require quarterly testing of PFAS in residuals for AOS holders (unless otherwise approved). However, due to the key recommendations of the MassDEP PFAS in residuals report and since field duplicates are used to assess both sample and lab variability, MassDEP is reducing the required frequency of testing Field Duplicates to one or two times per year.

Also, MassDEP will no longer be approving additional laboratories to perform PFAS analysis according to EPA Method 537 (modified by the laboratory as necessary). PFAS testing may be performed by [a laboratory with a MassDEP approved modified lab method](#), or by any laboratory using the draft method 1633 without MassDEP approval. This requirement might change in the future to require AOS facilities to test according to Method 1633 but this is not the case at this time.

Method Information- Revised

Analysis of selected per- and polyfluoroalkyl substances (PFAS) in residuals (i.e., biosolids) from facilities that distribute/sell residuals in Massachusetts under an Approval of Suitability (AOS) can only be performed by a laboratory approved by MassDEP to perform this analysis.

General Information

Please note that this revised procedure supersedes the February 8, 2021 and August 2020 MassDEP correspondence to all AOS holders. All AOS holders are required to test residuals quarterly for the following PFAS:

PFAS	Abbreviation	CASRN
Perfluorobutanoic acid	PFBA	375-22-4

Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTTrDA	72629-94-8
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335.77.335

Facilities must also submit all PFAS data generated according to the requirements of the U.S. EPA or other states.

Laboratory PFAS testing of residual samples shall include analysis of the solid product using appropriate analytical and quality control methods. PFAS testing results using the approved methodologies shall be submitted to MassDEP. Please submit results to eDEP according to instructions available here: <https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals>.

A list of laboratories approved by MassDEP for testing residuals for PFAS is maintained here: <https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals>

In preparation for sampling, AOS holders may want to review the following PFAS sampling guidance documents:

From the State of Michigan:

[https://www.michigan.gov/documents/pfasresponse/General PFAS Sampling Guidance 634597 7.pdf](https://www.michigan.gov/documents/pfasresponse/General_PFAS_Sampling_Guidance_634597_7.pdf)

From NEBRA:

[https://static1.squarespace.com/static/54806478e4b0dc44e1698e88/t/5ca2599a002291000145dd88/1554143647366/NEBRA-PFASSamplingAnalysisGuide v.2-5Jan2018.pdf](https://static1.squarespace.com/static/54806478e4b0dc44e1698e88/t/5ca2599a002291000145dd88/1554143647366/NEBRA-PFASSamplingAnalysisGuide_v.2-5Jan2018.pdf)

Laboratory Requirements for Testing Residuals for PFAS

Residual sample collection and testing for PFAS must be performed according to the following guidelines:

1. For residual PFAS analysis, laboratories are expected to extract residual samples according to ASTM Method D7968 (modified by the laboratory as necessary) with subsequent extract PFAS analysis according to EPA Method 537 (modified by the

laboratory as necessary). A laboratory must confirm that it meets a Minimum Reporting Level (MRL) for all target PFAS of less than or equal to 1.0 ng/g dry weight using spiked blank Ottawa sand (100% dry weight) as per the procedure in Section 9.1.4 of EPA Method 537. Also, a laboratory must minimize dilution of residual samples to only that which is necessary to handle significant matrix interference.

2. MassDEP expects that the facility will provide “Primary” and “Field Duplicate” residual samples (each with unique field and laboratory IDs) to the laboratory and that the laboratory will test both of these samples for PFAS using the laboratory's analytical method SOP approved by MassDEP for residuals PFAS analysis. The Field Duplicate must be sampled once or twice a year.
3. As you collect samples for PFAS testing, please be sure to include the following information in the Chain Of Custody that you send to the lab with your sample(s):
 1. Your NPDES ID number (included below)
 2. Indicate “Influent”, “Effluent”, “Sludge”, or “Industry” (for SIU indirect dischargers) for each sample
 3. For SIU industry samples, include the industry name(s) for each sample

This information will ensure that the lab adequately uploads information to the MassDEP PFAS website. If you are interested, the public portal that includes this information for SWD/NPDES and Residuals facilities is available to the general public and available (<https://eeaonline.eea.state.ma.us/portal#!/search/npdes>). Note that recent data might not yet be included in the public portal due to internal quality control.

4. MassDEP expects that with an extraction batch of 20 or fewer residual samples from one or more clients, the laboratory will select at least one of the residual samples as the Matrix Spike (MS) and MS Duplicate (MSD) or Laboratory Duplicate (LD) to be extracted, analyzed, and reported with the PFAS results for each of the residual samples in the extraction batch. MassDEP does not expect a facility to pay for these required QC samples even if the laboratory uses the facility's own residual sample for these QC analyses. AOS facilities must provide adequate sample amount for either the primary or field duplicate residual sample in case the MS and MSD or LD in an extraction batch is tested using that sample from an AOS holder. With the understanding that the amount of sample necessary varies depending on the aqueous nature of the residual, the amount of additional sample provided to the laboratory may vary and may include more than one container of the primary and/or field duplicate sample(s); note that the additional sample containers must be labeled with the same field and laboratory IDs as the corresponding primary or field duplicate sample.
5. An AOS holder may choose but is not required to submit a field reagent blank (FRB) with the primary and field duplicate residual samples to meet MassDEP's requirements for testing PFAS in residuals. An FRB is a reagent water sample that is transferred from one

bottle to a clean bottle at a PFAS sampling site and is therefore exposed to the field environment – it is used to determine if PFAS samples could have been contaminated in the field during the sampling process. Note that a trip blank (i.e., a reagent water sample in a sealed bottle that travels to the field and then back to the laboratory unopened with the field samples) is not necessary for PFAS analysis.

6. MassDEP expects that laboratory reports detailing PFAS in residuals will include analytical data for all applicable MS and MSD or LD performed in the same extraction batch(es) (as described above) as the residual samples. Note that MassDEP (currently in consultation with Tetra Tech) is reviewing each laboratory report of residual PFAS analysis. Providing acceptable QA/QC data will be critical to the approval of residuals PFAS analytical data.

If you have any questions regarding the MassDEP residuals program/AOS holders, please contact the Residuals Program at MassDEP.Residuals@mass.gov.

Resources Online

EPA Details of Method 1633: <https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas>

List of laboratories approved to test according to EPA Method 537 (modified by the laboratory as necessary: <https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals>

MassDEP Instructions regarding uploading PFAS results to eDEP: <https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals>

Public Portal for MA SWD/NPDES and Residuals facilities data: <https://eeaonline.eea.state.ma.us/portal#!/search/npdes>

Sampling Guidance from the State of Michigan: https://www.michigan.gov/documents/pfasresponse/General_PFAS_Sampling_Guidance_634597_7.pdf

Sampling Guidance from NEBRA: [NEBRA-PFASSamplingAnalysisGuide_v.2-5Jan2018 \(squarespace.com\)](https://www.nebra.gov/NEBRA-PFASSamplingAnalysisGuide_v.2-5Jan2018/squarespace.com)