

Department of Environmental Protection

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

From: MassDEP Residuals Program

Date: October 11, 2024

In accordance with 310 CMR 32.00 "Land Application of Sludge and Septage", the Massachusetts Department of Environmental Protection (MassDEP) is authorized to issue an Approval of Suitability (AOS) to any person who uses, sells, or distributes sludge or septage for land application in Massachusetts. Unless approved otherwise, MassDEP requires quarterly testing of PFAS in residuals for AOS holders.

This revised procedure document supersedes previous MassDEP correspondence to all AOS holders pertaining to laboratory testing for PFAS.

Definition of Terms

"Approval of Suitability" or "AOS" is approval from MassDEP for residuals to be used, sold, or distributed or offered for use, sale, or distribution.

"Biosolids" Sewage sludge that has been processed for land application.

"Residuals" are a broader term for sludge used for land application that includes biosolids. Other sludge included as "residuals" includes the product derived from drinking water treatment, processing of paper, and the processing of cranberries.

Summary of Changes

PFAS sampling conducted by all AOS holders requires laboratory analysis using EPA Method 1633 for the 40 PFAS Analytes listed in Attachment A. AOS holders shall transition to testing for the 40 Analytes using Method 1633 by **April 25, 2025**, unless required sooner by their AOS.

Method Information

Analysis of selected per- and polyfluoroalkyl substances (PFAS) in residuals covered by an AOS must be performed by a laboratory that is capable of performing EPA Method 1633 as

This information is available in alternate format. Please contact Melixza Esenyie at 617-626-1282. TTY# MassRelay Service 1-800-439-2370 MassDEP Website: www.mass.gov/dep demonstrated by its laboratory SOP documenting compliance with all Method 1633 analytical procedures, including all quality control requirements, and successful participation in annual single-blind solid (i.e., soil or biosolid) proficiency tests (PTs) for the analysis of target PFAS by Method 1633. Documentation shall be kept on file and available for MassDEP review and confirmation if so requested.

General Information

All AOS holders are required to test residuals quarterly for the PFAS analytes listed in Attachment A.

Laboratory PFAS testing of residual samples shall include analysis of the solid product using EPA Method 1633 solid matrix analytical procedures for target PFAS and meeting all method-specified quality control requirements. PFAS testing results using the approved methodologies shall be submitted to MassDEP noting any qualified results. Submit results to eDEP according to instructions available here: <u>https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals</u>.

In preparation for sampling, MassDEP recommends AOS holders review the following PFAS sampling guidance documents:

From the State of Michigan:

https://www.michigan.gov/documents/pfasresponse/General PFAS Sampling Guidanc e 634597 7.pdf

From NEBRA:

https://static1.squarespace.com/static/54806478e4b0dc44e1698e88/t/5ca2599a00229 1000145dd88/1554143647366/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. At least once a year the facility will provide both "Primary" and "Field Duplicate" samples (each with unique field and laboratory IDs) to the laboratory and that the laboratory will test both of these samples for PFAS.
- 2. 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):
 - a. The name of the facility holding the AOS
 - b. "Primary" or "Duplicate" for each sample, or "Other" for blanks
 - c. Method 1633
 - d. A note to the lab requesting that they upload data to eDEP

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 (<u>https://eeaonline.eea.state.ma.us/portal#!/search/npdes</u>). Note that recent data might not yet be included in the public portal due to internal quality control.

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

Resources Online

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

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

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 6345 97 7.pdf

Sampling Guidance from NEBRA: <u>NEBRA-PFASSamplingAnalysisGuide v.2-5Jan2018</u> (squarespace.com)

Details on Testing Method Requirements for PFAS testing of Residuals before October 11, 2024

When MassDEP first started requiring AOS facilities to test for PFAS in 2020, there was no EPAapproved method for testing residuals for PFAS, MassDEP therefore established a process to ensure that a laboratory would use appropriate methods and quality control. Therefore, MassDEP reviewed and approved the laboratory's proposed methodologies, including detailed Standard Operating Procedures (SOPs) for PFAS testing and the Initial Demonstration of Capability Certification (IDOC) Statements.

Attachment B includes details on the laboratories that applied for review, whose results were acceptable, and were approved for the testing of PFAS in residual products. MassDEP does not endorse the use of any one laboratory.

Attachment A- PFAS Compounds

Target Analyte Name	Abbreviation	CAS Number
Perfluoroalkyl carboxylic acids		
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	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluoroalkyl sulfonic acids		
Acid Form		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentansulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Fluorotelomer sulfonic acids		
1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4
1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2
1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4
Perfluorooctane sulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols		
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2
Per- and Polyfluoroether carboxylic acids		
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
4, 8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3, 6-dioxaheptanoic acid	NFDHA	151772-58-6

Target Analyte Name	Abbreviation	CAS Number
Ether sulfonic acids		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9CI-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Fluorotelomer carboxylic acids		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2H, 2H, 3H, 3H-Perfluorooctanoic acid	5:3FTCA	914637-49-3
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4

Attachment B- MassDEP Laboratories Approved for Testing of PFAS in Residuals before October 11, 2024

Lab Name: Alpha Analytical, Inc.

Address: 8 Walkup Drive, Westborough, MA 01581 Web Address: www.alphalab.com Contact: Jeremy Thebodo Email: jthebodo@alphalab.com Phone: 774-666-0360 SOP Date: 12/13/2019 MassDEP Approval Date: 2/10/2020

Lab Name: Contest, A Pace Analytical Laboratory

Address: 39 Spruce Street, East Longmeadow, MA 01028 Web Address: www.contestlabs.com Contact: Tod Kopyscinski or Katherine Allen Email: tkopy@contestlabs.com or kallen@contestlabs.com Phone: 413-525-2332 SOP Date: 2/26/2021 MassDEP Approval Date: 3/18/2021

Lab Name: Enthalpy Analytical, LLC.

Address: 2714 Exchange Drive, Wilmington, NC 28405 Web Address: https://enthalpy.com/ Contact: Lindsay Boone Email: lindsay.boone@enthalpy.com Phone: 910-544-6077 SOP Date: 4/30/2019 MassDEP Approval Date: 2/10/2020

Lab Name: Eurofins Lancaster Laboratories Environmental

Address: 2425 New Holland Pike, Lancaster, PA 17601 Web Address: https://www.eurofinsus.com/environment-testing/laboratories/eurofins-lancasterlaboratories-environmental/ Contact: Jane Huber Email: JaneHuber@eurofinsus.com Phone: 717-209-1438 SOP Date: 11/11/2019 MassDEP Approval Date: 2/11/2020

Lab Name: Eurofins Test America

Address: 880 Riverside Pkwy, West Sacramento, CA 95605 Web Address: https://www.eurofinsus.com Contact: Debby Wilson E-mail: Debby.Wilson@Eurofinset.com Phone: 949-237-0603 SOP Date: 5/9/2018 MassDEP Approval Date: 6/28/2019

Lab Name: Bureau Veritas Canada (2019) Inc. operating as Bureau Veritas Laboratories Address: 6740 Campobello Rd, Mississauga, ON, L5N 2L8 Web Address: https://www.bvna.com Contact: Salima Haniff E-mail: Salima.Haniff@bvlabs.com Phone: 905-817-5703, ext. 7065703 SOP Date: 3/25/2019 MassDEP Approval Date: 6/15/2020