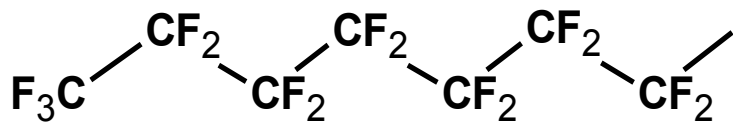


*NEBRA Guidance:*

# Sampling and Analysis of PFAS in Biosolids and Associated Media v. 3.2

June 2017 (v. 1.0), updated January 2018 (v. 2.0), June/July 2019 (v. 3.1.1), and August 2020 (v. 3.2)



## Acknowledgements

This guidance was written and produced by Michael Rainey, M.S. (Northwood, NH) with review and editing by members of the NEBRA PFAS Advisory Group and staff. Special thanks to New England Interstate Water Pollution Control Commission (Lowell, MA) for reliance on their biosolids sampling guide and to Professor Linda S. Lee, Ph.D., Purdue University (West Lafayette, IN).

This document is available at <https://www.nebiosolids.org/pfas-sampling-analysis-guidance>

Michigan EGLE published extensive guidance on PFAS sampling in 2018. See: [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)

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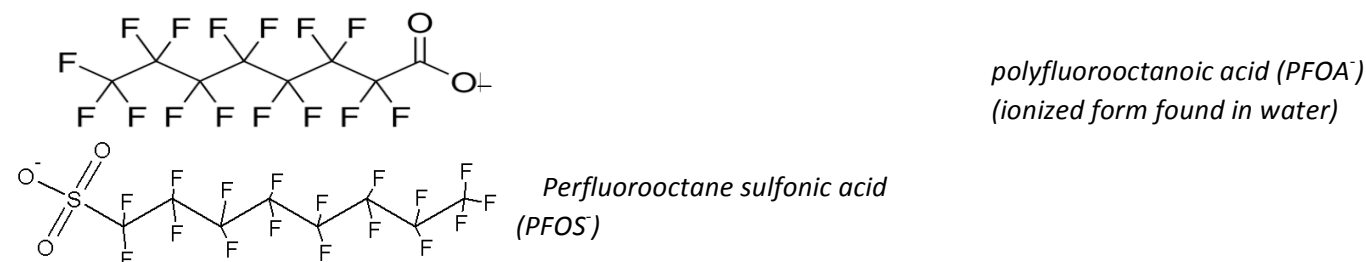
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## I. Introduction/Background

The U.S. Environmental Protection Agency (U. S. EPA) and state environmental agencies have shown increased interest and concern over the distribution of per- and polyfluoroalkyl substances (PFAS) in the environment. Contamination of drinking water has been a particular concern, especially with regards to the most ubiquitous and studied PFAS: PFOA and PFOS. As U. S. EPA and states have encountered increasing evidence of PFAS contamination in groundwater, especially drinking water sources, they are addressing potential health concerns. Pressure from the public and state legislatures has resulted in further attention and regulatory actions.

Since 2015, in the Northeast, regulatory agencies have addressed the most critical areas of concern surrounding known industrial sources of PFAS. In 2017, they expanded their areas of investigation from drinking and groundwater to other media and potential secondary conveyors of PFAS contamination, such as biosolids and other residuals (e.g. recycle paper mill residuals). Regulatory requirements pertaining to PFAS in wastewater residuals are changing rapidly. In 2019, Maine, Massachusetts, and New Hampshire initiated mandatory testing of wastewater residuals for PFAS. NEBRA became aware of this regulatory concern and recognized the challenges of obtaining robust data when the contaminants being analyzed are ubiquitous in society and the concentration levels of concern are in the ng/kg (parts per trillion, or ppt) range. (1 ppt = 1 second in ~32,000 years.) Sample contamination is a significant concern. Therefore, as environmental agencies, NEBRA members, and other professionals managing biosolids and other residuals investigate these sources, a thoughtful and measured approach is the most likely to provide useful information with the most efficient use of resources.

Any environmental investigation involving sampling of environmental media (biosolids, residuals, soil, water, etc.) should begin with the development of a *sampling plan*. A well-conceived sampling plan provides the best opportunity to produce accurate and defensible data for decision-making. Sampling to demonstrate regulatory compliance, or in response to regulatory inquiries, requires a sampling plan that incorporates the requirements and/or concerns of the regulatory agency or other investigator. At this stage, understanding of PFAS in the environment is still limited. Therefore, a collaborative effort between biosolids generators and managers, industry groups, regulators, and other interested parties that leverages the existing knowledge base to develop sampling plans and investigative strategies is a prudent approach to addressing the PFAS problem. This document is a step in that direction.



## II. Sampling Plans

A sampling plan is a step-by-step outline for how a sampling event or entire program will be implemented and performed. A comprehensive sampling plan should allow someone unfamiliar with sampling processes to read the plan and then collect samples that produce useable, accurate data.

Fortunately, an operator-friendly guidance document on how to develop a sampling plan already exists for wastewater and biosolids professionals. In 2006, the New England Interstate Water Pollution Control Commission (NEIWPCC) published “*The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans*,” it is available for free download in PDF format ([http://www.neiwpcc.org/neiwpcc\\_docs/biosampleguide/biosampleguide\\_web.pdf](http://www.neiwpcc.org/neiwpcc_docs/biosampleguide/biosampleguide_web.pdf)). Use of the NEIWPCC guide can help an operator develop a detailed sampling plan for PFAS testing of biosolids or sludge (solids). This NEBRA guidance relies heavily on the NEIWPCC guide by using its approach, format, and work sheets. However, guidance in this document will be specific to sampling and testing for PFAS. Generators and managers of residuals are encouraged to consult both this guide, as well as the NEIWPCC guide, if they choose to prepare a PFAS sampling plan.

### A. Developing a PFAS Sampling Plan

To start your PFAS sampling plan, obtain a photocopy or electronic copy of the Sampling Plan Worksheet in Appendix A. This work sheet was written primarily as a tool for municipal wastewater facilities sampling biosolids, but it can be adapted to a variety of sampling scenarios involving other environmental media. The worksheet has ten sections that need to be completed to produce a comprehensive sampling plan. Guidance on how to complete the worksheet is provided below. The worksheet itself has references to the NEIWPCC guide, which may provide a more thorough understanding of what is required to complete a specific section of the worksheet.

#### **Sections 1 & 2. Facility Information and Contact Person**

Sections 1 and 2 are self-explanatory; they provide basic facility and contact information.

#### **Section 3. Sample Plan Objective(s)**

It is important to state clearly the goals of the proposed sampling project. This section need not be lengthy, but it is important. A quick, one-time sampling event with limited objectives can be implemented utilizing a brief sampling plan. A long-term sampling project with multiple goals and sampling multiple environmental media (biosolids, residuals, soil, and water) will require a more detailed sampling plan. Clearly articulated goals will make it easier to complete the remaining sections of the worksheet. The goal statement can be as simple as acknowledging the need to comply with new regulatory requirements.

#### **Section 4. Facility Information**

This section provides basic information about the generating facility and its solids/biosolids/residuals production. All the information requested may not be pertinent to sampling events involving, for example, soils at land application sites or private wells, but much of the information is relevant to solids/biosolids/residuals quality and can provide

significant background data for any sampling scenario. Of particular interest is information on industrial or commercial discharges to the facility that may contain PFAS. Industries that use fluorosurfactants to produce outdoor clothing, stain-resistant fabrics, carpeting, food packing, fire-fighting foams, and cooking utensils are examples of potential PFAS dischargers.

## **Section 5. Data Quality Objectives**

Data quality objectives are established to ensure that data generated by sampling are adequate to meet the stated sampling program objectives. An in-depth discussion of data quality objectives and QA/QC is included in Chapter 5 of the NEIWPCC guide and will provide operators a working knowledge of these concepts. This guide focuses on the following four factors listed below to ensure appropriate data quality:

- 1) Qualified laboratory,
- 2) Approved analytical methods,
- 3) Required analytes, and
- 4) Adequate detection/reporting limits.

*Qualified laboratory:* As of August 2020, there are several commercial laboratories with a few years of experience in analyzing for PFAS. Ideally, you should use only environmental laboratories accredited by the state certifying agency or National Environmental Laboratory Accreditation Program (NELAP) for the matrix being sampled (solids, drinking water, etc.). However, there are still few accrediting authorities offering accreditation for PFAS, and there are no U. S. EPA-approved methods for PFAS analysis of solids. N.H. ELAP, administered by the New Hampshire Department of Environmental Services (NHDES), offers accreditation for U. S. EPA Method 537 Rev. 1 in drinking water and for specific laboratory procedures (modifications of Method 537) in other matrices. To help with lab procurement, Appendix B provides a list of labs that appear to have the instrumentation and expertise to provide reliable PFAS analytical services. State agencies sometimes also offer lists of laboratories that offer PFAS analysis, and some state agencies will have specific requirements regarding the lab(s) used. Check with your state agency.

*Approved analytical methods:* U. S. EPA has promulgated two analytical methods (U. S. EPA Methods 537 Rev. 1 (November 2018) and Method 533 (December 2019) exclusively for the analysis of PFAS. These methods as written are for the analysis of drinking water only and should be performed only with limited modification as allowed by the method. (Ensure Method 537 is updated to incorporate Technical Advisory EPA 815-B-16-021 published in September 2016 pertaining to PFOA analysis.) ASTM Method 7968 has been developed and validated for analysis of PFAS in soils. ASTM Method 7979 has been developed and validated for analysis of PFAS in non-drinking water, wastewater, and sludge. However, it may be difficult to find labs that utilize the ASTM methods. Various labs have modified Method 537 and added a variety of extraction protocols to accommodate testing of solids. If contracting with a lab using a modified version of Method 537, it is important to verify that the lab is producing high quality, defensible data. In late June, 2019, U. S. EPA released a draft Method 8327 for non-drinking waters, including wastewater, under the SW-846 program; it will be finalized by 2021. Appendix C-2 includes a discussion of the concerns associated with PFAS analysis and provides detailed guidance on how to evaluate the labs performing PFAS analysis and analytical data. The Department of Defense (DOD) has published a Quality Systems Manual for environmental analyses that provides detailed and specific criteria for evaluating PFAS analytical methods. Finally, research has shown that analysis of an aliquot of the sample can result in low-biased results. Labs should prepare and analyze the entire sample collected. Appendix C lists analytical requirements established

by northeast states, including analytical method requirements. See also NEBRA webpage on PFAS analysis for additional updates:  
<https://www.nebiosolids.org/pfas-sampling-analysis-guidance>.

*Required analytes:* For most environmental monitoring, the required target analyte list is usually determined by the state environmental agency or U. S. EPA. PFAS is a large class of chemicals, and in the recent past regulatory agencies were generally only interested in monitoring for a small subset of these chemicals (e.g. PFOA and PFOS). However, that has changed as state agencies, especially in the Northeast, have expanded target analyte lists to include more compounds and sometimes including all the analytes determined by Method 537.1 or more. Appendix C-1 provides target analyte lists required by states of the Northeast. U.S. EPA's Unregulated Contaminant Monitoring Rule (UCMR 3) lists six PFAS for monitoring by Method 537 (see Appendix C). If no list is provided for your state, it is recommended that analyses include PFOA and PFOS, at a minimum.

*Adequate detection/reporting limits:* U. S. EPA analytical methods, such as Method 537.1, include procedures for laboratories to determine their method detection limits and reporting limits. However, if a lab's reporting limits are at concentrations higher than the regulatory standard or guideline (e.g. U. S. EPA's public health advisory of 70 ppt for PFOA and PFOS separately or combined), then analytical data from that lab has no value in making comparisons to advisory/screening levels or for demonstrating regulatory compliance. Consequently, it is important to determine if the applicable regulatory agencies have established required detection limits or health-based, regulatory standards/guidelines and only contract with labs that can meet those requirements. Appendix C-1 is a compilation of state standards and required detection limits, if any, for the Northeast states. When analyzing solids (biosolids, residuals, etc.), NEBRA recommends working with labs that can confirm that they can achieve approximately 1 ug/kg (ppb) PFAS on a dry weight basis for residuals with a solids content down to as low as 10%.

Appendix C-1 summarizes current information pertaining to state requirements relevant for establishing data quality objectives (to the best of our knowledge, at the time of publication of this guidance). The current regulatory climate involving PFAS is rapidly evolving. It is advisable to contact state regulators to verify the most recent analytical requirements.

Finally, the Data Quality Objectives Section of sampling plan should include any requirements for field quality control/quality assurance (QA/QC) samples (trip blanks, rinsate blanks, etc).

## **Section 6. Sampling Points**

This section of the work sheet describes the selected sampling points and the rationale for their selection. For wastewater treatment facilities, the most appropriate sampling location is after treatment for pathogen and vectors. Biosolids and other wastewater residuals (e.g. paper mill residuals) should be sampled in the form in which they will be used. For sampling of soils at land application sites or private wells near a site, this section of the worksheet can be used to identify the location of the sampling site as well as the rationale for sampling at each location. This section of the sampling plan can also be used to provide background about sampling locations that may be helpful for interpreting analytical data. For example, is there a fire station or training facility in the neighborhood where fire-fighting foams may have been used, or were particular PFAS-containing products used on site in the past?

## **Sections 7 & 8. Sampling Collection and Handling Procedures**

These sections of the sampling plan describe the actual sampling procedure and handling of samples after collection. Descriptions of sampling collection and handling procedures are

frequently termed “standard operating procedures” or SOPs. Appendix D provides three examples of SOPs for three different sampling scenarios:

- 1) Sampling biosolids/residuals from a continuously dewatering/treatment process,
- 2) Sampling drinking water from a private well, and
- 3) Sampling surface soils (0-6 inches) from a biosolids/residuals land application site.

Some of the elements in these example SOPs are generic for any sampling procedure, and others are specific to the matrix being sampled. All three SOPs cover the sample collection process and some of the post-sampling handling procedures. Generators may need to modify an SOP if they are sampling a different matrix or sampling a similar matrix in a different way. For example, sampling surface water or groundwater monitoring wells requires a different SOP than that used for sampling drinking water from a private well. If a generator proposes to sample groundwater monitoring wells or sample soils at depths greater than 6 inches, they may consider contacting an experienced consultant familiar with these more complex sampling procedures. Also, these SOPs assume delivery of samples directly to the laboratory. If samples need to be stored and shipped, procedures for storage and shipping should be included in the SOP.

Appendix D also includes a list of general concerns for any PFAS sampling project, as well as a generic procedure for cleaning and decontamination of PFAS sampling equipment. Current groundwater and drinking water standards, as well as recently proposed standards, are set at extremely low concentrations (ppt). Since some PFAS are still used in commerce and are ubiquitous in the environment, contamination of samples from extraneous sources is a major concern for any sampling project. Appendix D-4 includes a table listing potential sources of PFAS contamination. Each of the SOPs and generic procedures include references that can be consulted for background information or to assist in the modification of the SOP.

## **Section 9. Evaluation of Completeness**

In this section of the sampling plan, generators can establish criteria for evaluation of their sampling program. For one-time sampling events, the only evaluation necessary is to determine if the goals of the sampling project were met and if additional sampling is necessary. For long-term sampling programs or programs with multiple goals, additional evaluation may be advisable. For example, do SOPs need to be revised? Were data quality objectives met? Did the lab provide usable and defensible data?

## **Section 10. Recordkeeping and Reporting**

For routine monitoring, state and federal biosolids regulations prescribe recordkeeping and reporting requirements. Although there are few requirements for recordkeeping or reporting of PFAS data, more states are requiring testing, and, therefore, reporting of these analytical results will be required. Generators required to perform PFAS testing should include any specific PFAS reporting requirements (units, timing, etc.) in this section. This section of the sampling plan can also be used to describe any specific data handling procedures specific to the facility’s or other organization’s PFAS sampling program. Note: Be sure to discuss with the lab and note in records that results are reported on a “dry weight basis” (or, alternatively, on an “as is” (wet weight) basis.

## References

Technical Brief: Per- and Polyfluoroalkyl Substances (PFAS), EPA/600/F-17/022. U.S. EPA. March 2017. [https://cfpub.epa.gov/si/si\\_public\\_record\\_report.cfm?dirEntryId=336072](https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=336072)

Technical Advisory - Laboratory Analysis of Drinking Water Samples for Perfluorooctanoic Acid (PFOA) Using EPA Method 537 Rev. 1.1, EPA 815-B-16-021. U.S. EPA, Office of Ground Water and Drinking Water, Cincinnati, OH 45268. September 2016.

The Third Unregulated Contaminant Monitoring Rules (UCMR 3): Fact Sheet for Assessment Monitoring (List 1 Contaminants), EPA 815-F-16-003. U.S. EPA, Office of Water (MS-140). May 2016.

Interim Guideline on the Assessment and Management of Perfluoroalkyl and, Polyfluoroalkyl Substances (PFAS), Department of Environment Regulation, Western Australia, January 2017. [https://www.der.wa.gov.au/images/documents/your-environment/contaminated-sites/guidelines/Guideline\\_on\\_Assessment\\_and\\_Management\\_of\\_PFAS\\_v2.1.pdf](https://www.der.wa.gov.au/images/documents/your-environment/contaminated-sites/guidelines/Guideline_on_Assessment_and_Management_of_PFAS_v2.1.pdf)

Perfluorocarboxylic Acid Content in 116 Articles of Commerce, EPA/600/R-09/033. 2009. [https://cfpub.epa.gov/si/si\\_public\\_record\\_report.cfm?dirEntryId=206124](https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=206124)

*The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plans*. New England Interstate Water Pollution Control Commission, Lowell, MA 01852-1124. September 2006. [http://www.neiwpcc.org/neiwpcc\\_docs/biosampleguide/biosampleguide\\_web.pdf](http://www.neiwpcc.org/neiwpcc_docs/biosampleguide/biosampleguide_web.pdf)

***Analyzing PFAS in Wastewater, Solids, & Soils: State of the Science Webinar, NEBRA Webinar, Sept. 14, 2017***

## Appendix A

# Sampling Plan

Facility / Site / Operation: \_\_\_\_\_

Sampling Plan Name: \_\_\_\_\_

Author(s) of Sampling Plan: \_\_\_\_\_

Contact Person (if other): \_\_\_\_\_

Date of Creation of Sampling Plan: \_\_\_\_\_

Date of Completion of Sampling Program: \_\_\_\_\_



# SAMPLING PLAN WORKSHEET

(Adapted from NEIWPCC's *The Wastewater Treatment Plant Operators Guide to Biosolids Sampling Plan*)  
(Please provide attachments as needed)

---

## 1. General Facility Information:

Facility Name:

Phone: (    )

Street Address:

City:

State:

Zip:

## 2. Contact Person:

Name:

Title:

Phone: (    )

Street Address:

City:

State:

Zip:

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## 3. Sampling Plan Objective(s):

(For explanation, see above and Chapter 3 of the NEIWPCC *Guide*.) Provide a statement that describes the goals of the sampling program and what media are to be sampled (drinking water, groundwater, wastewater solids, biosolids, soil, etc.).

## 4. Facility Information:

(See Chapter 4 of the NEIWPCC *Guide*.)

A. Provide a brief general description of your facility. For short-term soil or well sampling, this section may not be relevant and can be skipped. (Example: conventional activated sludge treatment with anaerobic digestion)

B. Design Flow (MGD): \_\_\_\_\_ Average daily flow (MGD): \_\_\_\_\_  
Previous Year's Annual Solids Production (dry metric tons): \_\_\_\_\_

C. Briefly describe the screening, grit removal, and flow equalization process employed at your facility.

*D. Describe the industrial pretreatment program, including a list of permitted facilities, especially if they are potential dischargers of PFAS.*

*E. Describe any treatment processes (such as advanced treatment for nutrient removal) that may affect solids quality, if any.*

*F. Describe the source and generation of solids. Do the solids contain primary solids? How are solids stored, both before and after dewatering? What is the dewatering method and what chemicals are used in the dewatering process?*

*G. How are the solids treated to achieve pathogen reduction and vector attraction reduction?*

**5. Data Quality Objectives:** (See above and/or Chapter 5 of the NEIWPCCC *Guide* and Appendix D below.)

*A. List the PFAS analytes for which testing is required or desired.*

*B. Specify the analytical methods required for the sample matrix to be collected. This may be determined by the state regulatory agency requesting the testing.*

*C. Specify the detection/reporting limits needed for this sampling project. Again, state regulatory agencies may specify detection/reporting limits based on the matrix being sampled. Detection/ reporting limits should always be at concentrations less than any regulatory standard or guideline.*

*D. What type of samples will be collected (grab or composite)? If a composite sample is collected, how many grab samples will be collected and what will be the interval between grabs? What will be the sample size?*

**6. Sampling Points:** (See above and/or Chapter 6 of the NEIWPCCC *Guide*.)

*Provide a detailed description of all sampling points along with the rationale for their selection. If sampling a private well or soils from agricultural field, identify the location and rationale for sampling at the location.*

**7. Sample Collection Procedures:** (See above and/or Chapter 7 of the NEIWPCCC *Guide*.)

*Please provide a detailed standard operating procedure (SOP) describing the process used for collecting samples. The step-by-step description should include all details pertaining to sample collection, including a description of the cleaning and preparation procedures for sampling equipment and sample containers. **Examples of SOPs for three different matrices are included in Appendix D, below.***

**8. Sample Handling Procedures:** (See Chapter 8 of the NEIWPCCC *Guide* and/or consult with lab.)

*Describe the post-collection sample handling procedures employed to maintain sample integrity. This description should explain how the samples will be preserved and transported, what the appropriate hold-time is for each analysis, and whether a chain-of-custody is required. SOPs can be edited to include post-collection sampling handling procedures.*

**9. Evaluation for Completeness:** (See above and/or Chapter 9 of the NEIWPCCC *Guide*.)  
*Describe the process to be used for evaluating the completeness of the sampling effort. Criteria for evaluation might include: Were the goals of the sampling program met? Were data quality objectives achieved? Do the data quality objectives or SOPs need to be revised?*

**10. Record-Keeping and Reporting:** (See Chapter 10 of the NEIWPCCC *Guide*)  
*Provide a description of record-keeping procedures. The description should explain what information will be retained and for how long, how the information will be stored, and what records are required to be reported, if any.*

## Appendix B. Laboratories Providing PFAS Analytical Services updated August 11, 2020

Laboratory	Contact Name	e-mail	Address	Phone	Accreditation
Alpha Analytical	Steve Knollmeyer	<a href="mailto:Sknollmeyer@alphalab.com">Sknollmeyer@alphalab.com</a>	8 Walkup Drive Westborough, MA 01581	(603) 498-7213 (Steve) or 800-624-9220 (lab)	ME, NH, VT, etc.
ALS Global	Chris Leaf	<a href="mailto:Chris.Leaf@alsglobal.com">Chris.Leaf@alsglobal.com</a>	1317 South 13th Ave Kelso, WA 98626	(360) 577-7222	
Enthalpy Analytical	Laura Boivin	<a href="mailto:laura.boivin@enthalpy.com">laura.boivin@enthalpy.com</a>	2714 Exchange Drive Wilmington, NC 28405	(910) 520-2526	MA, etc.
Eurofins Lancaster Laboratories Environmental, LLC	Jane Huber or Charles Neslund	<a href="mailto:JaneHuber@eurofinsUS.com">JaneHuber@eurofinsUS.com</a> <a href="mailto:charlesneslund@eurofinsus.com">charlesneslund@eurofinsus.com</a>	2425 New Holland Pike, Lancaster, PA 17601	(717) 556-1438 (Jane cell) (717) 556-7231 (Chuck cell)	MA, ME, etc.
Eurofins Test America	Debby Wilson	<a href="mailto:Debby.Wilson@Eurofinset.com">Debby.Wilson@Eurofinset.com</a>	880 Riverside Parkway, West Sacramento, CA 95605	(916) 373-5600 (lab)	MA, NH, etc.
SGS Axys Analytical Services	Nick Corso	<a href="mailto:nicholas.corso@sgs.com">nicholas.corso@sgs.com</a>	2045 Mills Road West, Sidney B.C. V8L 5X2 Canada	781-799-5740 (Nick cell) (888) 373-0881 (lab)	CA, LA, NJ, FL, MN, AN, AB, etc.
Vista Analytical	Jennifer Christman	<a href="mailto:info@vista-analytical.com">info@vista-analytical.com</a>	1104 Windfield Way El Dorado Hills, CA 95762	916-995-5171 (Jennifer cell) (916) 673-1520 (lab)	NH, MI, etc.

**\*This table may be updated as new information becomes available. Contact NEBRA: [info@nebiosolids.org](mailto:info@nebiosolids.org) or 603-323-7654**

## Appendix C-1. Northeast States' Standards and Analytical Requirements (as of June 1, 2017\*)

State	Target Analytes	Sludge/Biosolids			Drinking Water/Groundwater			Soil		
		Standard (ug/kg)	Required DL (ug/kg)	Method	Standard (ng/L)	Required DL	Method	Standard (ug/kg)	Required DL	Method
Connecticut	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorononanoic acid (PFNA) Perfluorohexanesulfonic acid (PFHxS) Perfluoroheptanoic acid (PFHpA)	N/A	N/A	N/A	70 - total conc. of PFOA PFOS PFNA PFHxS PFHpA	N/A	N/A	N/A	N/A	N/A
Maine	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorobutanesulfonic acid (PFBS)	PFOA - 2.5 PFOS -5.2	Below std.	Isotope dilution method with at least 10% rec of isotopes	N/A	N/A	N/A	PFOA -2.5 PFOS -5.2	Below std.	Isotope dilution method with at least 10% rec of isotopes
Massachusetts	Perfluorobutanoic Acid (PFBA) Perfluoropentanoic acid (PFPeA) Perfluorohexanoic acid (PFHxA) Perfluoroheptanoic acid (PFHpA) Perfluorooctanoic acid (PFOA) Perfluorononanoic acid (PFNA) Perfluorobutanesulfonic acid (PFBS) Perfluorohexanesulfonic acid (PFHxS) Perfluorooctanesulfonic acid (PFOS)	N/A	N/A	Requires SPLP as well as solid sample analysis	70 - total conc. of PFOA PFOS PFNA PFHxS PFHpA	N/A	EPA Method 537 or 537.1	N/A	N/A	N/A

New Hampshire	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorononanoic acid (PFNA) Perfluorohexanesulfonic acid (PFHxS) Perfluoroheptanoic acid (PFHpA) Perfluorobutanesulfonic acid (PFBS) Perfluorobutanoic acid (PFBA) Perfluoropentanoic acid (PFPeA) Perfluorohexanoic acid (PFHxA)	N/A	advising that 1 ppb is acceptable	Modified 537 by isotope dilution	Final (effective 10/1/2019)  PFHxS -18 PFNA -11 PFOS - 15 PFOA - 12	2 ng/L	Analytical methods that use isotope dilution conducted by laboratories accredited by the Department of Defense or the National Environmental Laboratory Accreditation Program (NELAP); or 2) EPA Method 537 Rev 1.1 following the provisions of EPA Technical Advisory 815-B-16-021 issued in September 2016 for PFOA by laboratories with NELAP accreditation from NH or another state.	500 ug/Kg	N/A	NELAP accreditation from NH or another state for solids analysis
New Jersey	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorononanoic acid (PFNA)	N/A	N/A	N/A	PFNA - 13 Proposed PFOA -14 PFOS -13	3 ng/L PQL	Method 537 Rev 1.1	N/A	N/A	N/A
New York	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS)	N/A	N/A	N/A	70 ng/L for PFOS and PFOA combined	unknown	Method 537 Rev 1.1	N/A	N/A	N/A
Rhode Island	No regulatory requirements	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Vermont	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorononanoic acid (PFNA) Perfluorohexanesulfonic acid (PFHxS) Perfluoroheptanoic acid (PFHpA)				20 - total conc. of PFOA PFOS PFNA PFHxS PFHpA	unknown	Method 537 Rev 1.1	300 ug/kg	N/A	N/A

U.S. EPA-UCMR 3	Perfluorooctanoic acid (PFOA) Perfluorooctanesulfonic acid (PFOS) Perfluorononanoic acid (PFNA) Perfluorohexanesulfonic acid (PFHxS) Perfluoroheptanoic acid (PFHpA) Perfluorobutanesulfonic acid (PFBS)	N/A	N/A	N/A	70 ng/L for PFOS and PFOA combined (health advisory)	20 ng/L 40 ng/L 20 ng/L 30 ng/L 10 ng/L 90 ng/L	EPA Method 537 Rev 1.1 following the provisions of EPA Technical Advisory 815-B-16-021 issued in September 2016 for PFOA	N/A	N/A	N/A
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DL=detection limit, RAGS= Maine’s Remedial Action Guidelines, PQL=practical quantitation limit, UCRM 3= EPA’s Third Unregulated Contaminate Monitoring Rule

**\*This table may be updated as new information becomes available. Contact NEBRA: [info@nebiosolids.org](mailto:info@nebiosolids.org) or 603-323-7654.**



## APPENDIX C-2

### Analysis of PFAS – Details for the Lab Consumer

#### Biosolids, Soils, Wastewater, and Other Matrices Other Than Drinking Water

December 2017; minor updates June 2019 and August 2020

#### The Current State of Lab Analysis of PFAS

Measuring PFAS – a family of chemicals with a variety of structures – is challenging. However, samples have been collected and analyzed in a wide variety of media for many years, and there are research papers reporting analytical methods and data from analyses. Currently, there seems to be widespread agreement that liquid chromatography tandem mass spectrometry (LC/MS-MS) is the best and most common analytical tool.

U. S. EPA has promulgated two analytical methods: U. S. EPA Method 537 Rev.1 (November 2018) and Method 533 (December 2019) exclusively for the analysis of PFAS. These methods as written are for the analysis of drinking water only and should be performed without modification. Be sure the lab is using the latest version, Method 537 Rev. 1 (November 2018) and/or Method 533. They are complementary and cover an overlapping list of analytes.

Currently, commercial and other labs have each developed what they call “modified Method 537.” These are isotope dilution methods, which the U. S. Department of Defense (DoD) prefers. However, none of these are U. S. EPA-approved, and the modifications vary from lab to lab. Each lab writes its own Standard Operating Procedure (SOP) for its modified Method 537, and staff of some large, reputable labs believe that, when best industry standards are followed, the modifications developed by different labs are likely very similar. In addition, some labs have participated in multi-lab comparison quality control studies to determine the accuracy of data resulting from different labs’ modified Methods 537. We have not reviewed those inter-lab studies, but have been told that some labs perform well and others don’t. Because of the competitive business environment, obtaining additional information is challenging. Data on a multi-lab blind sample comparison reported by William Lipps of Shimadzu in 2017, working with U. S. EPA, indicated considerable variation in data quality from participating commercial labs using their various “modified Method 537.”

ASTM Method D7968 has been developed and validated for analysis of PFAS in soils. ASTM Method D7979 has been developed and validated for analysis of PFAS in non-drinking water, including influent, effluent, and sludge. For screening purposes, if low detection limits are not required, the ASTM methods would be the preferred analytical protocols for PFAS in soil, wastewater, or wastewater residuals. In the multi-lab comparison reported above, they performed well. However, it may be difficult to find labs that utilize the ASTM methods. They do not achieve as low detections as the “modified Method 537” being used by most labs, which relies on isotope dilution and standards.

Additional updates on lab analytical methods can be found at: <https://www.nebiosolids.org/pfas-sampling-analysis-guidance>.

#### Data Quality Concerns

Remember, you are the client, the customer of the lab. Shop for a reputable lab wisely, as you would for any product you spend substantial money on. It is okay to ask questions and expect helpful answers.

As noted above, various labs have modified Method 537. Expert chemists at U. S. EPA and elsewhere have expressed some concerns about the quality of data being produced by these various modified methods, including:

- **unusually wide surrogate recovery ranges of 25% - 150%** - More standard is a range of 70% to 130%. Wider ranges decrease accuracy when the reportable concentration is adjusted based on surrogate recovery.
- **limited number of surrogates available and in use** - This issue continues to diminish, as more commercial

surrogates are available for labs to purchase.

- **use of aliquots** – Many labs will only use a portion of a sample they receive; research has indicated that this may misrepresent PFAS concentrations. Best practice is to analyze the entire sample received or extracted.
- **labs using single point calibration rather than calibration curves**
- **short LC/MS run times** – U. S. EPA uses run times in the 16 – 21 minute range. Quality control data from some reputable commercial labs indicates run times of 4 – 5 minutes. Short run times make it more difficult to separate out individual compound peaks from matrix interferences.
- **the data quality varies for different PFAS** - Reviews of lab reports indicate significant differences in the quality of chromatographs and data for different individual PFAS.
- **loss of short-chain PFAS during sample concentration** – compounds of lower molecular weight may be more volatile and subject to loss during concentration of sample extracts.

## Recommendations for Working With Labs on PFAS Analysis

It is important to verify that the lab is producing high-quality, defensible data. Advice for working with a contracted laboratory is provided below.

### 1. Before sampling and contracting with a lab:

- Learn what method the lab uses and discuss quality control with them.
- Ask for details on the lab's standard operating procedure (SOP) for their PFAS method and quality control (QC) procedures, whether they have taken part in multi-lab quality control comparisons, whether they do ongoing method performance checks in real matrices, and if they will provide you with complete quality control data, including all blanks and chromatographs, for some or all of the analyses they complete for you. They may charge extra for what is called a "Level 4" QA/QC report, which will be a PDF of hundreds of pages. It is useful to ask about its availability, should you want it.
- Discuss with the lab what forms of PFOS and other compounds (e.g. PFHxS, PFBS) they measure and report. PFOS can refer to several similar compounds that have different CAS numbers. Obtain the CAS numbers for the compounds analyzed and be sure they match your sampling and analysis plan and any regulatory or screening standards.
- Be sure to get the lab's recommendations on sampling protocols, containers to use, sample sizes, field and other blanks, sample storage and shipping, and schedules.
- Discuss how the results will be reported. The norm is for data to be in units of mass of PFAS per mass of media on a dry weight basis. Ensure the units and "dry weight basis" appear in writing on test results.

### 2. When you get the results:

- Ask for the full quality control package for the analyses they completed for you. Most labs provide a quality control summary that is 10 – 30 pages. That is helpful, but for at least a few of your tests, you want the data behind that summary - the PDF with hundreds of pages. (Get it electronically. It may cost a bit extra.) When you ask the lab for it, specify that you want the following information and associated quality control data for each analysis or group of analyses and any other quality control systems that help validate and calibrate your test results:
  - Any standard documents that communicate QA/QC information to their clients
  - Reporting limits
  - Detection limits
  - Data on analyses of method, field, and other blanks
  - Data on laboratory control samples (blank spikes)
  - Laboratory duplicate relative percent difference (RPD)
  - Matrix spike recoveries
  - Matrix spike duplicate recoveries
  - Surrogate recoveries
  - Isotope recoveries

- Internal standard recoveries
- Method reporting limit checks
- Chromatographs of all the sample analyses, as well as of all blanks, calibrations, and calibration checks
- Did they use the whole sample? How extensive were sample manipulations?
- How much did they rely on isotope dilution? Did they dilute out isotopes and have to add more isotopes? How much time was allowed for equilibration of the isotopes? Were the isotopes at a similar concentration as the data reporting range?

Ask lab staff to at least introduce you to the data package and show you what each section of the report is showing you. If they are willing, ask them to walk you through it in more detail. You can also consider having the package reviewed by an independent party. NEBRA can help with this.

3. When reviewing the data quality package, pay close attention to the following:

- Ensure the data are reported on a mass of PFAS per mass of media on a dry weight basis.
- The narrative/notes page – summarizes any quality control issues that the lab identified
- Look at the volumes of the sample they used. Did they use the whole sample? How much did they prepare and how much did they inject into the LC/MS?
- How long were the run times? Longer - up to 20+ minutes - is better.
- Look at chromatographs: are the peaks smooth and clear, or are they raised, misshapen, and overlapping, creating blobs? How did the lab interpret these to report a concentration number? Did they just ignore some of the “blobby” chromatographs or report them as non-detects?
- Are ion ratios used and shown? Different single reaction monitorings (SRMs)?
- Look at the data and chromatographs for the field and method blanks. Are the peaks and data clean and simple? They should be.
- EPA provides detailed guidance on data validation of organic analyses in National Functional Guidelines for Superfund Organic Methods Data Review, EPA-540-R-2016-002, September 2016. It can be downloaded from the EPA website ([https://www.epa.gov/sites/production/files/2016-09/documents/national\\_functional\\_guidelines\\_for\\_superfund\\_organic\\_methods\\_data\\_review\\_0.pdf](https://www.epa.gov/sites/production/files/2016-09/documents/national_functional_guidelines_for_superfund_organic_methods_data_review_0.pdf))

**Lists of Specific PFAS Included in Standard Analytical Methods**

MDL = minimum detection limit

***EPA-Approved Method 537 Rev. 1.1***

Method Detection Limit and Single Lab Minimum Reporting Limit (Table 5, Method 537)

Analyte	MDL (ng/kg)	Reporting Limit (ng/kg)
PFBS	3.1	3.7
PFHxA	1.6	2.9
PFHpA	0.5	3.8
PFHxS	2.0	8.0
PFOA	1.7	5.1
PFNA	0.7	5.5
PFOS	1.4	6.5
PFDA	0.7	3.8
NMeFOSAA	6.5	14

NEtFOSAA	4.2	14
PFUnA	2.8	6.9
PFDoA	1.1	3.5
PFTTrDA	2.2	3.8
PFTA	1.7	4.7

**ASTM D7968 (PFAS in soil):**

Method Detection Limit and Reporting Ranges

Analyte	MDL (ng/kg)	Reporting Limit (ng/kg)
PFTreA	6.76	25–1000
PFTriA	5.26	25–1000
PFDoA	3.56	25–1000
PFUnA	2.45	25–1000
PFDA	5.54	25–1000
PFOS	18.83	50–1000
PFNA	2.82	25–1000
PFecHS	2.41	25–1000
PFOA	6.24	25–1000
PFHxS	7.75	25–1000
PFHpA	5.80	25–1000
PFHxA	15.44	50–1000
PFBS	6.49	25–1000
PFPeA	20.93	125–5000
PFBA	22.01	125–5000
FHEA	199.04	600–20 000
FOEA	258.37	750–20 000
FDEA	137.46	500–20 000
FOUEA	4.85	25–1000
FhpPa	5.09	25–1000
FHUEA	3.50	25–1000

**ASTM D7979 (PFAS in water, sludge, influent, effluent, wastewater):**

Method Detection Limit and Reporting Ranges

Analyte	MDL (ng/kg)	Reporting Limit (ng/kg)
PFTreA	1.2	10 – 400
PFTriA	0.7	10 – 400
PFDoA	1.2	10 – 400
PFUnA	1.2	10 – 400
PFDA	1.4	10 – 400
PFOS	2.2	10 – 400
PFNA	1.1	10 – 400
PFecHS	1.9	10 – 400
PFOA	1.7	10 – 400
PFHxS	1.2	10 – 400
PFHpA	1.0	10 – 400
PFHxA	2.0	10 – 400
PFBS	0.8	10 – 400
PFPeA	4.6	50 – 2000
PFBA	4.6	50 – 2000
FHEA	92.9	300 – 8000
FOEA	106.8	300 – 8000
FDEA	47.2	200 – 8000
FOUEA	2.3	10 – 400
FHpPA	3.3	10 – 400
FHUEA	1.5	10 – 400

## **Appendix D. Standard Operating Procedures (SOPs)**

Appendix D-1. General Concerns for All PFAS Sampling Projects

Appendix D-2. SOP: Biosolids/Residuals/Solids Sampling

Appendix D-3. SOP: Soil Sampling, 6-inch Topsoil Layer

Appendix D-4. SOP: Private Drinking Water Well Sampling

Appendix D-5. Equipment Preparation and Cleaning Procedure for PFAS Sampling

## D-1. General Concerns for All PFAS Sampling Projects

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PFAS are ubiquitous in modern society and the environment; their use in consumer products is still common (although, as of 2015, PFOA and PFOS have been mostly phased out of use in the U. S. and Canada and are now generally only in older products and some imported products).

Drinking water and groundwater advisory levels and regulatory standards for PFOA and PFOS are extremely low, and analytical methods are very sensitive, with detection limits in the low nanogram per liter (ng/L) or part per trillion range (ppt). The pervasive presence of these chemicals, coupled with very sensitive analytical methods, makes contamination of samples both in the lab and in the field a significant concern. Solids (biosolids, residuals, soils, etc.) sampling and analysis, while not as sensitive (typical concentrations are in the low microgram per kilogram (ug/kg) or parts per *billion* (ppb)), are still susceptible to PFAS contamination from sources extraneous to the environmental media being sampled.

**The following recommendations and concerns should be considered when undertaking any PFAS sampling program.**

- 1. There are many consumer products and materials that can contaminate samples** during the collection and handling process. The sampler should attempt to keep these materials away from the sampling area and avoid physical contact with anything likely to contain PFAS, including food, clothing, and personal care products. The N.H. Dept. of Environmental Services has published a useful document entitled “PerFluorinated Compound (PFC) Sample Collection Guidance;” below is an excerpted and updated table from that document that details items that should be avoided when sampling for PFAS.
- 2. It is preferable to use sample containers provided by the laboratory** that will perform the analyses requested. These containers generally will be polypropylene or HDPE. Components of the sample containers, such as lid liners, should not contain Teflon or polytetrafluoroethylene (PTFE).
- 3. All sampling equipment that will contact the sample should be cleaned** according to the equipment cleaning and preparation protocol in Appendix D-5.
- 4. Wash hands and use new nitrile gloves (powderless are best) to collect each sample.** The gloves should not contact the sample media, if possible, and should be changed if their cleanliness is compromised.
- 5. Avoid contacting the inside of the sample container or cap** with anything other than the material being sampled.
- 6. When samples have been collected and capped, they should be sealed in an individual ziplock bag** and placed in a cooler with only ice.
- 7. If collecting samples for analysis for other parameters,** the container for PFAS should be filled *first* and stored in a separate cooler for transport to the lab.
- 8. Samples of areas with low PFAS concentrations should be sampled before areas known to be highly contaminated.**
- 9. One member of the sampling team should collect the sample, and another perform other functions** such as labeling bottles, taking field notes, or other activities that may increase the potential for contamination of the sample.
- 10. It is important to include field QC such as field blanks, trip blanks, and equipment rinsate blanks in the sampling program** to ensure collection procedures and decontamination procedures are effective and are not introducing extraneous PFAS contamination into samples

## Items Prohibited and Allowed at PFAS Sampling Locations

Category	Prohibited Items	Allowed Items
Pumps and Tubing	Teflon® and other fluoropolymer containing materials, pipe thread seal tape	High-density polyethylene (HDPE), low density polyethylene (LDPE), or silicone tubing, peristaltic pump or stainless-steel submersible pump
Decontamination	Decon 90	Alconox® or Liquinox®, potable water followed by deionized rinse.
Sample Storage and Preservation	LDPE or glass bottles, PTFE-or Teflon®-lined caps, chemical ice packs, aluminum foil	Laboratory-provided sample container <i>preferred</i> ; or, HDPE or polypropylene bottles, regular ice sealed in plastic (polyethylene) bags to prevent melt water contaminating samples, thin HDPE sheeting
Field Documentation	Waterproof/treated paper or field books, plastic clipboards, non-Sharpie® markers, Post-It® and other adhesive paper products	Plain Paper, <i>metal</i> clipboard, Sharpies® (allowable per EPA, but other markers are not), pens
Clothing	New or unwashed clothing, clothing or boots made of or with Gore-Tex™ or other synthetic water resistant and/or stain resistant materials, coated Tyvek® material, anything washed with fabric softeners.	Well-laundered synthetic or 100% cotton material, previously laundered clothing (preferably previously washed greater than six times) <i>without</i> the use of fabric softeners. Steel-toed or other boots made with polyurethane and/or polyvinyl chloride (PVC). <i>Uncoated</i> Tyvek.
Personal Care Products (for day of sample collection)	Cosmetics, moisturizers, hand cream, some sunscreens, insect repellants, and other related products, dental floss and plaque removers	Suncreens: Alba Organics Natural Yes to Cucumbers Aubrey Organics Jason Natural Sun Block Kiss My Face Insect Repellents: Jason Natural Quit Bugging Me Repel Lemon Eucalyptus Herbal Armor California Baby Natural Bug Spray BabyGanics Sunscreen and Insect Repellents: Avon Skin So Soft Bug Guard-SPF 30
Food and Beverage	Pre-packaged food, fast food wrappers or containers, aluminum foil, non-stick cookware & containers	Bottled water or hydration drinks.

### References

- 1) PerFluorinated Compound (PFC) Sample Collection Guidance. N.H. Dept. of Environmental Services. November 2016. <https://www.des.nh.gov/organization/commissioner/documents/pfas-sample-guidance-201611.pdf>
- 2) Soil Sampling, SESDPROC-300-R3. Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. Aug. 21, 2014.
- 3) Groundwater Sampling, SESDPROC-301-R3. Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. March 6, 2013.
- 4) Dora Chiang, Ph. D., P. E., AECOM, PFAS Sampling. Presentation to NEWMOA, August 3, 2016. [http://www.newmoa.org/events/docs/228/PFAS\\_Sampling\\_Chiang\\_Aug2016.pdf](http://www.newmoa.org/events/docs/228/PFAS_Sampling_Chiang_Aug2016.pdf)



## **D-2. Standard Operating Procedure: Biosolids/Residuals/Solids Sampling**

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1. A week to several days prior to the proposed sampling, confirm solids (sludge) processing (dewatering and treatment) to ensure that solids in the appropriate form (liquid versus dewatered, untreated cake versus treated biosolids) will be available for sampling at the proposed date, time, and sampling point.
2. A week to several days prior to the proposed sampling date, confirm that the contract laboratory performing the analyses is prepared to accept samples on the proposed sampling date.
3. At least one day before collecting samples, assemble the sampling equipment. Ensure that all equipment is clean and in good working order (see attached checklist and appended cleaning procedure (Appendix D-5)).
4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.
5. After arrival at the sampling location/sampling point (as determined in the sampling plan), evaluate the operation of the solids handling train (dewatering, biosolids treatment, etc.). Any observable deviations from normal operation should be noted prior to collecting samples.
6. Put on nitrile gloves and any other required/desired personal safety equipment.
7. Collect a single grab sample using a stainless-steel trowel. Collect the sample from the belt filter press, conveyor or other accessible process as the solids are transported to the hauling vehicle. Fill the sample container provided by the lab with the recommended amount of sludge (usually 2 to 5 ml). Do not forget to collect any required field duplicates, blanks, equipment blanks or other field QC.
8. Upon collection of the sample, record the date and time and label the container with the information listed in #9 below.
9. Label all sample containers with the following information:
  - a) Sample identification number (ID)
  - b) Date and time of collection
  - c) Sample location
  - d) Person collecting sample
  - e) Preservative method (typically ice and chilling to 4°C for solids)
  - f) Required test(s)
10. After labeling, seal the sample container with a signed custody seal, if necessary, and place in ziplock bag which will keep the container clean and dry. Preserve all samples on ice (0-6°C) for transportation to the laboratory
11. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.
12. After sample delivery, clean all equipment according to established procedures (see Appendix D-5) and store in a clean, dry area.

### **References**

- 1) *The Wastewater Treatment Plant Operators Guide To Biosolids Sampling Plans*. New England Interstate Water Pollution Control Commission, Lowell, MA. Sept. 2006.
- 2) *Field Sampling Quality Control*, SESDPROC-011-R4. Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. Feb. 5, 2013.
- 3) *Guidance on Choosing a Sampling Design for Environmental Data Collection*, EPA QA/G-5S, EPA /240/R-02/005. USEPA, Office of Environmental Information, Washington, DC 20469, Dec. 2002

## EQUIPMENT CHECKLIST – Biosolids / Residuals / Solids

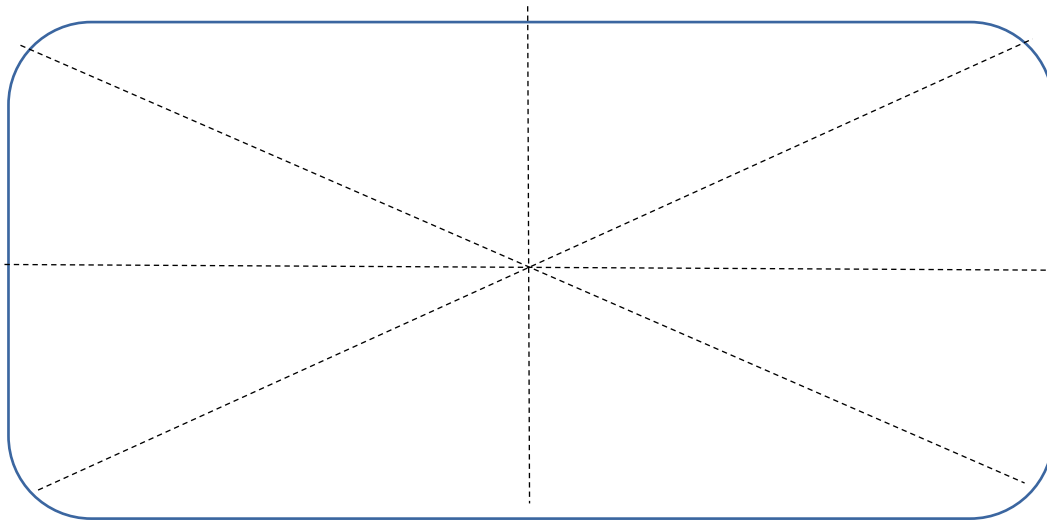
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- 1) Sample handling and collection
  - a. Nitrile gloves
  - b. Stainless steel bucket
  - c. Stainless steel trowel or lab spatula
- 2) Transporting and preservation
  - a. Sample containers provided by the lab
  - b. Sample cooler with ice
  - c. Ziplock bags
- 3) Sample ID and Documentation
  - a. Sharpie markers and pens
  - b. Custody seals
  - c. Chain of custody/sample submittal form
  - d. Sample log/field data sheet
- 4) Cleaning equipment
  - a. Disposable towels
  - b. Soap
  - c. Scrub brush
  - d. Tap water
  - e. Deionized water
  - f. Methanol
  - g. Plastic wrap
- 5) Ensure that all equipment, supplies, and other materials assembled for sampling, including clothing worn by sampling staff, will not contaminate samples with PFAS extraneous to the residuals being sampled. **See Appendix D-1 for materials that may contain PFAS and that should be avoided.**

### **D-3. Standard Operation Procedure: Soil Sampling, 6-inch Topsoil Layer**

1. A week to several days prior to the proposed sampling event, confirm the fields proposed for sampling will be available at the proposed date and time.
2. A week to several days prior to the proposed sampling date, confirm that the contract laboratory performing the analyses is prepared to accept samples on the proposed sampling date and arrange to receive the appropriate sampling containers, sample submittal forms, and mailing containers, if necessary.
3. At least one day before collecting samples, assemble the sampling equipment. Ensure that all equipment is clean and in good working order (see attached checklist and appended cleaning procedure (Appendix D-5)).
4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.
5. After arrival at the sampling location (as determined in the sampling plan), note briefly the weather conditions and any field conditions that might affect sampling results. For example, is any portion of the field flooded? Are there any areas of obvious erosion? Has the field been recently tilled or planted?
6. Put on nitrile gloves and any other required/desired personal safety equipment.
7. Collect a composite sample from an area of less than 5 acres by collecting 8-12 grab samples using a foot-step soil probe. If the field being sampled is greater than 5 acres, collect one composite for each 5-acre area:
  - a. It is recommended that an equal number of grab samples be collected randomly along each of four transects as shown schematically in Figure 1 below,
  - b. For each grab sample, the probe should be advanced to a depth of 6 inches then withdrawn, removing a 6-inch soil core which is then transferred to a stainless steel or HDPE bucket,
  - c. Grab samples are collected and accumulated in the bucket until the desired number of grab samples to form the composite sample has been reached.

**Figure 1. Schematic of Farm Field and Four Sampling Transects**



8. Upon collection of the last grab sample, thoroughly mix all soil accumulated in the stainless steel bucket with a stainless-steel trowel. The goal of the mixing process is to produce a homogeneous sample.
9. Label all sample containers with the following information:

- a) Sample identification number (ID)
- b) Date and time of collection
- c) Sample location
- d) Person collecting sample
- e) Preservative method (typically ice and chilling to 6°C for solids)
- f) Required test(s)

10. After labeling, fill the sample container provided by the laboratory with a portion of the homogenized material from the stainless-steel bucket. Generally, 2-5 g of sample will be sufficient for analysis.

11. After each sample container is filled, seal it with a signed custody seal and place it on ice in a cooler for transportation to the laboratory. Placing the sampling container in a ziplock bag, will keep the it clean and dry.

12. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.

13. Clean core sampling equipment according to established procedures (see Appendix D-5) between each composite sample site location. After sample delivery, clean all equipment according to established procedures (see Appendix D-5) and store in a clean, dry area.

## References

- 1) Soil Sampling. Midwest Laboratories, Inc. Omaha, NE 68144
- 2) Soil Sampling, SESDPROC-300-R3. Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. Aug. 21, 2014.
- 3) Description and Sampling of Contaminated Soils: A Field Pocket Guide, EPA/625/12-91/002. U. S. EPA, Center for Environmental Research Information, Cincinnati, OH, 45268. November 1991.

## EQUIPMENT CHECKLIST - Soil

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- 1) Sample handling and collection
  - a. Nitrile gloves (powderless are best)
  - b. Stainless steel bucket
  - c. Stainless steel trowel
  - d. Foot-step soil probe
- 2) Transporting and preservation
  - a. Sample containers provided by the lab
  - b. Sample cooler with ice
  - c. Ziplock bags
- 3) Sample ID and Documentation
  - a. Sharpie markers and pens
  - b. Custody seals
  - c. Chain of custody/sample submittal form
  - d. Sample log/field data sheet
- 4) Cleaning equipment
  - a. Disposable towels
  - b. Soap
  - c. Scrub brush
  - d. Tap water
  - e. Deionized water
  - f. Methanol
  - g. Plastic wrap
- 5) Ensure that all equipment, supplies, and other materials assembled for sampling, including clothing worn by sampling staff, will not contaminate samples with PFAS extraneous to the soil being sampled. **See Appendix D-1 for materials that may contain PFAS and that should be avoided.**

#### **D-4. Standard Operation Procedure: Private Drinking Water Well Sampling**

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1. A week to several days prior to the proposed sampling event, confirm the private well(s) proposed for sampling will be available on the proposed date and time.
2. A week to several days prior to the proposed sampling date, confirm that the contract laboratory performing the analyses is prepared to accept samples on the proposed sampling date and arrange to receive the appropriate sampling containers, sample submittal forms, and mailing containers, if necessary.
3. At least one day before collecting samples, assemble the sampling equipment. Ensure that all equipment is clean and in good working order (see attached checklist and appended cleaning procedure (Appendix D-5)).
4. On the day of sampling, obtain ice for sample storage and transportation and place in sample coolers.
5. After arrival at the sampling location note conditions that might affect sampling results. For example, is the well newly installed or recently decontaminated?
6. Wash your hands and put on nitrile gloves and any other required/desired personal safety equipment.
7. Collect samples in an area free of excessive dust, rain, snow or other sources of contamination.
8. Select a cold-water faucet for sampling which is free of contaminating devices such as screens, aeration devices, hoses, purification devices, swiveled faucets or plumbing where Teflon tape has been used. The tap off the pressure tank is recommended because it avoids other household plumbing. Check the faucet to ensure it is clean. If the faucet is in a state of disrepair, select another sampling location.
9. Collect samples from faucets which are high enough to put a bottle underneath, generally the bathtub or kitchen sink, without contacting the mouth of the container with the faucet.
10. Open the faucet and thoroughly flush by running the faucet for 5 minutes. Typically, the water temperature will stabilize, which indicates flushing is completed. Once the lines are flushed, adjust the flow so, it does not splash against the walls of the bathtub, sink or other surfaces.
11. Remove the lid of the sampling container, being careful not to touch the inside of the lid or container or allow them to contact other surfaces. Fill the container and screw the lid back on tightly.
12. Label all sample containers with the following information:
  - a) Sample identification number (ID)
  - b) Date and time of collection
  - c) Sample location
  - d) Person collecting sample
  - e) Preservative method
  - f) Required test(s)
13. After each sample container is filled, seal it with a signed custody seal and place it in a ziplock bag, which will keep the container clean and dry. Place all sampling in a cooler on ice for transportation to the lab.
14. Prior to delivering the samples to the lab, complete a chain-of-custody sheet to document proper sample handling.
15. After sample delivery, clean all equipment according to established procedures (see Appendix D-5) and store in a clean, dry area.

## References

- 1) Quick Guide to Drinking Water Sample Collection, 2<sup>nd</sup> ed. U. S. EPA, Region 8 Laboratory, Golden, CO, 80403. September 2016.
- 2) N.H. Sample Collection & Preservation Manual for Drinking Water, R-CO-01-5. NH Dept. of Environmental Services, Concord, NH 03302-0095. February 2001.
- 3) METHOD 537. DETERMINATION OF SELECTED PERFLUORINATED ALKYL ACIDS IN DRINKING WATER BY SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (LC/MS/MS), EPA/600/R-08/092. Version 1.1, September 2009.

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## EQUIPMENT CHECKLIST – Private Well

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- 1) Sample handling, collection, and preservation
  - a. Nitrile gloves (powderless are best)
  - b. 250 mL polypropylene bottle with Trisma preservative
  - c. Sample cooler with ice
  - d. Ziplock bags
- 2) Sample ID and Documentation
  - a. Markers and pens
  - b. Custody seals
  - c. Chain of custody/sample submittal form
  - e. Field notebook/ sample log/field data sheet
- 3) Cleaning equipment
  - a. Disposable towels
  - b. Soap
  - c. Tap water
  - d. Deionized water
  - f. Plastic wrap
- 4) Ensure that all equipment, supplies, and other materials assembled for sampling, including clothing worn by sampling staff, will not contaminate samples with PFAS extraneous to the water being sampled.  
**See Appendix D-1 for materials that may contain PFAS and that should be avoided.**

## D-5. Equipment Preparation and Cleaning Procedure for PFAS Sampling

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The following cleaning procedure should be used to clean all HDPE, polypropylene, stainless steel, or other equipment used to collect samples for PFAS analysis. Because of the extremely low detection and reporting levels required for PFAS analysis, precaution should be taken to ensure cleaning materials (soap, tap water, deionized water, methanol) are not contaminated with PFAS prior to use. It may be necessary to have cleaning materials analyzed (e.g. with a rinsate blank; see 7, below) to ensure they are not contaminated.

- 1) Rinse equipment with warm tap water to remove most solids.
- 2) Using a brush and a low-phosphate lab detergent, scrub the equipment to remove all residues. Liquinox® or Luminox® are common lab detergents that can be used for this purpose.
- 3) After scrubbing, rinse the equipment three times with tap water.
- 4) The tap water rinse should be followed by rinsing three times with deionized water (PFAS-free).
- 5) Finally, the equipment should be triple rinsed with methanol. The rinsate from this step should be collected for proper disposal with other organic chemical wastes. (As of spring 2019, Maine DEP prefers final rinsing with PFAS-free water.)
- 6) After cleaning, allow the equipment to air-dry. Many cleaning protocols suggest covering or wrapping clean sampling equipment in aluminum foil. U. S. EPA Method 537 advises against this practice to avoid possible contamination of the equipment with PFAS. This caution is particularly important for water samples. To store, buckets, beakers and other containers can be inverted in a clean, dry location. Soil probes, trowels, augers, and other sampling equipment should be covered or wrapped with clean, unused HDPE or polypropylene plastic sheeting.
- 7) Periodically – and especially at the beginning of a sampling project – equipment rinsate blanks should be collected. A rinsate blank is prepared by rinsing equipment previously cleaned (using the method outlined above) with deionized water and collecting the rinse water for analysis. Equipment rinsate blanks are particularly important when equipment is cleaned in the field between samplings.

Note: Because of the potential for PFAS cross-contamination, it is best to avoid field cleaning of sampling equipment, if at all possible.

### **References:**

- 1) METHOD 537. DETERMINATION OF SELECTED PERFLUORINATED ALKYL ACIDS IN DRINKING WATER BY SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (LC/MS/MS), EPA/600/R-08/092. Version 1.1, September 2009.
- 2) Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206-R3. Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. Dec. 18, 2015.
- 3) Field Sampling Quality Control, SESDPROC-011-R4, Region 4, U. S. EPA, Science and Ecosystem Support Div., Athens, Georgia. Feb. 5, 2013.