PFAS Sampling Standard Operating Procedure (SOP) for Wastewater Treatment Systems

Maine DEP ○ September, 2022

1.0 INTRODUCTION

This guidance documents covers the Standard Operating Procedure (SOP) for MEPDES wastewater treatment facilities to sample their effluent for perfluoroalkyl and polyfluoroalkyl (PFAS) compounds. PFAS sampling of monitoring wells is not covered in this document. Spray irrigation facilities who are required to collect PFAS samples from both a groundwater well and an effluent monitoring point can use this SOP for the effluent sample collection.

All PFAS samples will be GRAB SAMPLES. Following collection, samples must be analyzed by a Maine-accredited laboratory using EPA Method 537.1 Modified with Isotope Dilution. Results are reported for up to 28 individual PFAS compounds, as well as reporting Total PFAS comprised of the "sum of 6": perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), perfluorohexane sulfonic acid (PFHxS), perfluorononanoic acid (PFNA), perfluoroheptanoic acid (PFHpA) and perfluorodecanoic acid (PFDA). The standard reporting limit is 2 ng/L (nanograms per liter, a.k.a., parts per trillion or ppt), however a higher detection limit may be necessary for specific samples, such as those with high solids or matrix interferences.

This SOP covers potential sources of PFAS contamination, pre-sampling preparation, sampling equipment, collection, preservation, storage, quality control (QC), documentation, equipment decontamination (decon), and transport of samples to the contract lab for analysis

The DEP will continue to update this guidance as new information becomes available. For more information, refer to the DEP PFAS website, https://www.maine.gov/dep/spills/topics/pfas/.

2.0 POTENTIAL SAMPLE CONTAMINATION

PFAS compounds are present in many common household products, clothing, consumer packaging and personal care products. Given the ubiquitous nature of PFAS compounds and the low detection levels that are generally requested for analysis, more stringent precautionary steps are required to prevent PFAS cross-contamination when collecting samples when compared to routine sampling for common wastewater parameters.

Personal care products that may contain PFAS should be avoided on sampling days. These include moisturizers, sunscreen, insect repellent or similar products that have not been determined to be PFAS-free. Commercial and industrial products that may contain PFAS include water-proof clothing, coated paper, blue ice, and plastic clipboards. PFAS can be found in food packaging and certain foods and beverages. Sampling ports and equipment containing LDPE or Teflon® sealants, tapes, or tubing should not be used during PFAS sampling. All grab sampling equipment used in this wastewater study must be properly cleaned and decontaminated to help reduce cross contamination. See Table 1 for a summary of PFAS-containing products to avoid on PFAS sampling days.

Remember, it is in the best interest of the facility to reduce the potential for sample contamination to the greatest extent possible. Greater than actual results could lead to additional sampling and analysis events, or long term, burdensome permit limits and unnecessary remedial actions. Therefore, careful preparation and following procedures is critical for accurate, successful PFAS sampling at your facility.

Table 1. Personal Items to Avoid on PFAS Sampling Day

Items to Avoid on PFAS Sampling Day
● Avoid use of moisturizers, sunscreen, insect repellent or similar products that have not been determined to be PFAS-free on the day of the sampling event.
● Avoid prepackaged food or fast-food packaging on sampling day. No food should be eaten in the sampling or staging area.
● Do not wear on sampling day clothing and other materials that have been laundered with any chemicals, i.e. fabric softeners, waterproofing. Instead, use plain, unscented detergent or soap. New clothing should be washed several times before use.
● Examples of clothing or gear that may contain PFAS include Gore-Tex™, Tyvek®, Teflon®, any fire-retardant clothing, and certain Carhartt products (e.g. "rain defender", "DWR", "Rugged Flex", etc.).
● Keep PFAS sampling containers away from carpeting and upholstery. Do not use foil as a layer between stacked sample bottles.
● Do not use latex or vinyl gloves, pumps or tubing that contain Teflon® or other fluoropolymer-containing materials, or recycled or chemically treated paper towels.
● When performing field documentation, do not use waterproof/treated paper or field book, plastic clipboard, spiral bound notebook, permanent marker, Post-It® or other adhesive paper products.
● Do not use chemical (blue) ice packs to cool samples. Lab or commercially purchased ice is acceptable.
● Untreated paper towels and cotton cloths may be used during the sampling event.

3.0 Preparation Prior to Conducting PFAS Sampling

a) Sampling and Analysis Plan SAP). Prior to conducting any investigative field work, a sampling and analysis plan (SAP) should be developed. Elements of the sampling plan include names of personnel managing the project and collecting the samples, personnel training, sampling documentation, sample location, methods for collecting, storing and transporting samples to the contract lab, sample equipment decontamination and storage between sampling events, and receipt and maintenance of PFAS sampling results. See Attachment 1 for a SAP template.

b) Training. All personnel involved in PFAS sampling should be properly trained on PPE, proper sampling technique, equipment, completing required documentation, and determining which personal care products and consumer materials should be avoided on PFAS sampling days.

c) Gloves. Single use disposable powder-free nitrile gloves should be worn for the duration of the sampling event.

d) Clothing. A cotton or cotton blend lab coat or utility overall should be worn over street clothes that has been washed several times in fragrance- and softener-free laundry detergent prior to using the first time and washed again following each sampling event.

e) Consumer products. See Table 1 for a summary of PFAS-containing products to avoid on PFAS sampling days. Do not allow any prepackaged or fast-food packaging in the sampling area. No food should be eaten in the staging or sampling areas.

f) Sampling Documentation. PFAS sampling documentation includes Chain of Custody (COC), sample bottle labels, sampling logs, and/or field data sheets.

- The DEP and/or the contract lab will supply the required forms for data reporting. Information includes facility location and contacts, sampling location, sampling date and time, collection methods, and required signatures. For each participating public facility, these forms will be customized and provided as a template.

- The DEP may require field data sheets and/or sampling logs that include plant condition at the time of sampling, such as wet weather flow, septage received, and total daily flow. For each participating public facility this information is entered in a section provided on the customized COC.

- Private commercial/industrial facilities participating in the Project will be selecting their preferred Maine-certified analytical laboratory. Each participating facility will ensure that the COC used contains at least the same information as the state template.

g) Sampling Equipment. The facility must prepare to receive, store, and decontaminate (decon) sampling equipment using PFAS-free materials, such as HDPE or stainless steel. All equipment must be properly cleaned, rinsed with PFAS-free water, and stored in a clean location until the next sampling event. Equipment should be rinsed again with PFAS-free water prior to re-use.

- In most cases a dedicated cooler will be supplied by the contract lab. Included in the sampling "kits" are two, 250 mL sample bottles, bottle labels, COC forms, and Ziplock® bags for storing collected samples. The kit may also include Quality Control (QC) sample bottles (field blank [two], equipment blank [two], and a temperature blank), and approximately one liter of PFAS-free water for equipment decon. Sample bottles must be polypropylene with unlined, polypropylene caps (no Teflon®-lined caps).

- The coolers should only be used to store PFAS samples and equipment. The cooler may stay at the facility between sampling events or be used to transport samples to the lab, depending on the pickup/delivery plan for each location.

- Equipment used for sampling collection and any portion of dippers, long-handled swing samplers, scoop/extension rod, etc. that come into contact with the effluent sample should made of PFAS-free material, such as high-density polyethylene (HDPE) or stainless steel. Sample-taking containers should be a minimum of 1 liter capacity, and not greater than about 2 liters. If ropes are necessary for sample collection, use natural fiber ropes.

h) Determine PFAS sampling location(s). For required sampling, the DEP Compliance Inspector can assist the facility regarding the appropriate PFAS sampling location. In most cases PFAS samples will be collected at or near the same location as MEPDES final effluent samples, or as otherwise directed.

- Facilities without open access to the effluent stream or which are unable to collect a grab sample from a tap directly into an active wastewater discharge line must consult their DEP Compliance Inspector to determine an appropriate sampling location or collection technique.

- Facilities using ports or pumps for sampling collection must verify that submersible pumps, tubing, and bladders are PFAS-free (HDPE, silicone, or stainless steel). Use of pumps or other kinds of more complex sampling equipment will entail the use of equipment blanks.

- Avoid grabbing samples from areas containing high solids, excessive turbulence or foam, as PFAS compounds can accumulate on suspended solids and are attracted to the air/water interface of bubbles and foam.

i) Scheduling PFAS sampling. For required sampling, the DEP will notify the facility of the sampling schedule. ***In general, this will be once a month for 10 consecutive months for effluent samples, and once a quarter for a minimum of four samples for groundwater monitoring. Additionally, field blanks and equipment blanks will be required with the first two sampling events.*** Public facilities participating in the Project will be using Alpha Analytical to provide courier (sample kit delivery and sample pickup), analytical, and data acquisition (Level Report of analyses and EGAD-compatible EDD form completion). Participating commercial/industrial facilities who elect to use Alpha Analytical may be able to use the same services, as individually determined.

- Avoid times of upsets or unscheduled maintenance activities unless specifically requested by the DEP. Weather (rain, snow, wind, etc.) impacts your ability to accurately collect your effluent sample, consider rescheduling for another day to avoid cross contamination from water-proof rain gear or other causes. If necessary, use wet weather gear and boots made of polyurethane and PVC only.

- Facilities with intermittent discharges should contact their compliance inspector to determine when to perform PFAS sampling.

- Facilities where winter conditions prevent a Q1 2023 groundwater sampling event will alternatively run two samples in Q2 or Q3.

- As applicable, shipping arrangements with the contract lab for sample drop off/pick up should be made at least 48 - 72 hours prior to the sampling day. When properly packed in ice the sample hold time for Method 537.1 M is 14 days. However, it is advisable that samples be held no longer than a maximum of 2 – 3 days before shipment to allow for handling and transportation variability, weekends, and holidays.

j) Sampling Practices

- In most cases the sample bottle will contain a small amount of white powder (Trizma) to dechlorinate the sample. To avoid losing the Trizma, the effluent sample will be collected in an intermediate container and transferred to the sample bottle.

- The collection container used by the facility should be PFAS-free material (i.e., HDPE or stainless steel) with a volume of at least 1-L that was properly cleaned and rinsed with PFAS-free water. The contract lab should provide a container for this purpose.

- Any equipment used during sample collection that comes into contact with the effluent sample, such as long-handled swing samplers, scoop/extension rod, etc. should made of PFAS-free material. If ropes are necessary for sample collection, use natural fiber ropes.

- Samples collected from sampling ports (or pumps): Attach PFAS-free tubing or sampling equipment to sampling ports, pumps, etc., *if necessary*, in accordance with the sampling plan. Turn on the tap (or pump) and allow the effluent to flow freely for at least 5 minutes before collecting the sample to obtain a representative sample, free of potential local contamination.

k) Quality Control (QC) Samples. QC samples, including field and/or equipment blanks will be only collected when requested by DEP or the contract laboratory. The purpose is to check for air-borne, site, or sampling technique contamination. A temperature blank (a bottle of PFAS-free water) may be included by the lab to verify that the samples are properly cooled during transportation.

4.0 SAMPLING COLLECTION PROCEDURES

a) Collect a field blank: Two bottles are supplied by the lab; an empty bottle and a full bottle of PFAS-free water. Both bottles should be labeled “field blank”. At the start of the sampling event, open both bottles, pour the full bottle of PFAS-free water into the empty bottle and close. *Include both the full field blank bottle and the empty PFAS-free water bottle in the sample package being returned to the lab.*

b) Collect an equipment blank: Two bottles are supplied by the lab; 1 Trizma bottle (empty 250 mL) and 1 PFAS-free water (filled 250 mL). Both bottles should be labeled "equipment blank". Pass the PFAS-free water over or through the sampling device and into the empty Trizma 250 mL bottle that is labeled "equipment blank".

c) Collect the PFAS sample: Using a PFAS-free collection container, rinse with site medium several times. Collect a grab sample under the effluent stream. Carefully transfer the sample from the collection container into the sample bottles, filling two, 250 mL bottles. The bottles should be filled to the neck (or fill-line if present), with headspace left at the top.

d) Don't let the inside of the bottle, cap or threads touch anything. Do not place bottle caps on the ground during sampling or they may get contaminated. It is best to hold the bottle caps with one hand while sampling with the other. Get help from an appropriately prepared assistant if you need it.

e) Once filled, cap and gently flip bottle upside down (~5x) until the preservative (a small amount of white-powder, Trizma®, used for dechlorination) is mixed in. Make sure not to overfill the bottles or pour any water out in order to avoid losing powder. Once closed, do not re-open the bottle.

f) Affix the bottle labels and complete required information. Put the bottles into zip-lock bags and place in the cooler.

g) Complete the COC, noting sampling date, time, sampler name, and sign where necessary. Include any comments and other information required. Verify the date and time on the bottle labels match the COC. Put COC in plastic bag and place in the cooler. Public facilities participating in the Project will be using pre-labeled bottles and pre-filled COCs requiring date, time, and sampling individual inputs (COCs require additional information); it is advisable that writing on the sample bottles take place immediately ahead of the sampling activity. Commercial/industrial facilities should follow the same protocol.

h) Complete COCs or field data sheets as required by the DEP. This may include information on plant conditions at the time of the sampling, such as wet weather, septage received, and total daily flow.

i) All collected sample bottles and required paperwork should be put into Ziplock® plastic bags and stored in the dedicated cooler packed with regular ice from your lab or commercially purchased. Do not use "blue ice" chemical icepacks. Do not place any other non-PFAS samples in the same cooler. Make sure the temperature blank (a bottle of PFAS-free water) is included in the cooler.

j) Store coolers with collected samples in a safe location while maintaining the temperature ≤ 10°C (50°F) during the first 48 hours, and ≤ 6°C after 48 hours. Do not allow samples to freeze. If samples must be stored for a day or two while awaiting transfer to the lab, it may be possible to store smaller coolers in a designated refrigerator but avoid storing near other items that may contain PFAS. The preferred technique is to store the samples in ice in the cooler, replenishing the ice as needed.

k) Ship the coolers to the contract lab in accordance with the sampling plan. Adequate ice is particularly important when collecting samples during hot weather or for overnight shipment.

l) Properly discard disposable equipment. Do not reuse nitrile gloves. Decon reusable equipment in accordance with Section 5.0 below. Cover the clean equipment and store in a dedicated location for the next sampling event. Do not use the sampling equipment for non-PFAS sampling.

5.0 DECONTAMINATION (DECON)

a) Clean equipment and containers thoroughly with Alconox® or Liquinox® labware cleaner and tap water. Do not use Decon 90 or Dawn® dish detergent.

b) Perform a final rinse with PFAS-free water. In most cases, PFAS-free water will be supplied by the contract lab and should be reserved for the final rinse.

c) Store the clean sampling container in a Ziplock® bag, separate from the rope, line, or other equipment used. Do not use this sampling container or tie line for any purpose other than PFAS sampling.

6.0 PFAS TESTING RESULTS

PFAS lab results are typically available from the laboratory 3-4 weeks following sample receipt. Results include a Level 2 QC lab report and an Electronic Data Deliverable (EDD) form. For public facilities in the project, this has been pre-arranged with Alpha Analytical. Commercial/ industrial facilities should consult with their Compliance Inspector regarding reporting data. Information on how to read and interpret the PFAS lab report can be found at https://www.maine.gov/dep/water/wwtreatment/index.html.

Results are subject to QC review by DEP personnel prior to upload into the state's Environmental and Geographic Analysis Database (EGAD) system. Results will be available on the DEP PFAS website https://www.maine.gov/dep/spills/topics/pfas/ where other PFAS data is reported.

For more information, contact your DEP Compliance Inspector.

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Project Technical Advisor
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Table 2 - PFAS Effluent Sampling Project Contacts:

Maine DEP Project Managers:
James Crowley, DEP Compliance Supervisor, State Pretreatment Coordinator
207-287-8898
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Brett Goodrich, DEP Technical Assistance
207-287-9034
Brett.A.Goodrich@maine.gov
Judy Bruenjes, DEP Technical Assistance
207-287-7806
Judy.K.Bruenjes@maine.gov
ALPHA Analytical:
Michael Chang, Alpha Analytical Project Manager
Contact for CoCs, reports, EDDs or other sample issues
508-439-5124
320 Forbes Ave, Mansfield, MA 02048
mchang@alphalab.com
Steve Knollmeyer, Alpha Account/Sales Rep
Contact for account set up, capabilities, pricing, contracts
603-498-7213
72 Center Street, Brewer, ME 04412
sknollmeyer@alphalab.com
Cindy Rogers, Brewer Service Center Manager
Contact for sample containers, courier setup)
Cindy Rogers (crogers@alphalab.com)
207-624-0713
72 Center Street, Brewer, ME 04412
Billy Dean, Maine Courier
Contact for sample pickup/ cooler delivery
wdean@alphalab.com, 207-852-1397

Attachment 1 - Sampling and Analysis Plan (SAP) Template

Facility Name:	**Location:**
MEPDES #:	**DEP Compliance Inspector:**
Analytical Lab Used: **Contact email and phone #:**	
Name of person responsible for overseeing sampling project: **Contact email and phone #:**	
Person responsible for sample collection and communication with lab (if different): **Contact email and phone #:**	
Sampling Point Name (Outfall or Lagoon Effluent before Spray):	
Which describes your PFAS sample location? ☐ **Outfall pipe or cascade** ☐ **Spigot** ☐ **Sampling port** ☐ **Tank** ☐ **Lagoon effluent** ☐ **Other** _____	
Sample bottles will be filled: ☐ **Directly from a spigot** ☐ **Dip bucket or sampling device will be used**	
Check which one applies: ☐ **The facility has a continuous discharge. Samples will be available every month beginning in Oct 2022 thru July 2023.** ☐ **The facility has an intermittent discharge. Provide details on when you will be generating effluent for sample collection.**	

Have personnel involved in PFAS sampling been properly trained on PPE, proper sampling technique, equipment, completing required documentation, and determining which personal care products and consumer materials should be avoided on PFAS sampling days? ☐ **Yes** ☐ **No**
Describe the methods for collecting, storing, and transporting samples to the contract lab, sample equipment decontamination and storage between sampling events, and receipt and maintenance of PFAS sampling results.
The facility receives (check all that apply): ☐ **Septage** ☐ **Landfill leachate** ☐ **Other Transported Wastes** **How is this documented?**