

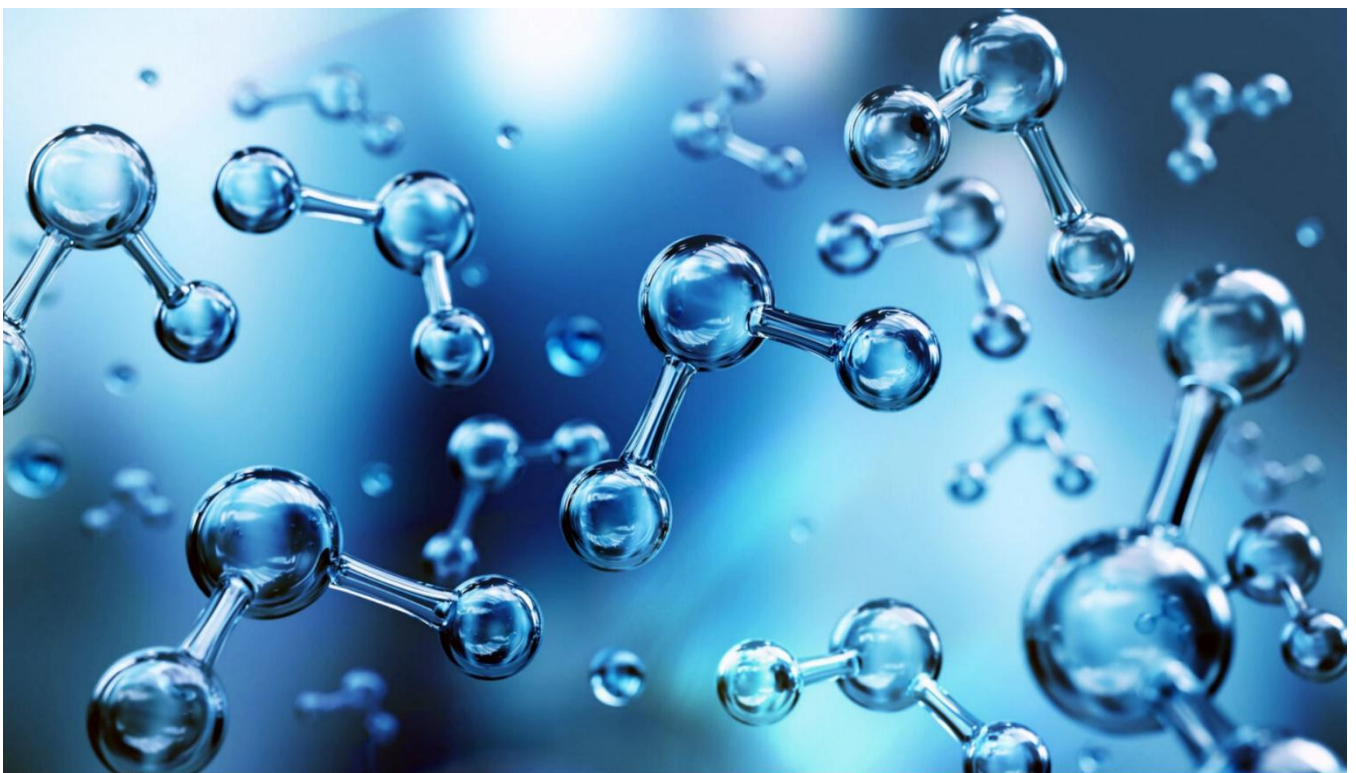


Maryland
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**Maryland Department of the Environment
Water and Science Administration
Per- and polyfluoroalkyl substances (PFAS) Sampling Guidance Document for
Wastewater Analysis**



Date: 02/06/2023

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Water and Science Administration
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EXECUTIVE SUMMARY

Per- and polyfluoroalkyl substances (PFAS) are man-made chemicals that have been used in industry and consumer products worldwide since the 1950s. These chemical compounds are now considered contaminants of emerging concern. **PFAS** have been linked to many harmful **health effects**, including cancer, immune system dysfunction, liver damage, developmental and reproductive harm, and hormone disruption. These compounds have been used in non-stick cookware, firefighting foam, water-repellent outerwear, stain-resistant fabrics, and products that resist grease, water, and oil.

The most frequently evaluated PFAS are perfluorooctanoic acid (**PFOA**) and perfluorooctane sulfonic acid (**PFOS**). PFOA and PFOS have been gradually phased out of production starting around 2000 and banned for use in the United States in 2014. The next most studied are perfluorohexane sulfonic acid (**PFHxS**), and perfluorononanoic acid (**PFNA**).

During the production of raw and final products and use, PFAS can enter the environment through the soil, water, and air. The majority of PFAS do not break down or biodegrade so they persist in the environment. Because of the widespread use of PFAS and their persistence in the environment, PFAS are found in the blood of humans and animals all over the world. In addition, PFAS are present at low levels in a variety of food products and in the environment. PFAS have been used for decades in many common products but their presence in the environment is still not completely understood because until now the toxicological and human health risks associated with PFAS have had limited assessments. Although our knowledge of the human health risks due to PFAS exposures is still deficient, exposures to these compounds have been associated with a wide range of adverse health effects.

OBJECTIVE

This *Sampling Protocols should be used by Maryland Permittees* when conducting sampling for PFAS analysis. The purpose of this document is to ensure the accuracy and uniformity of the analytical data reported and ensure that the data reported represent the actual discharge concentrations of PFAS at Maryland's WWTPs and other selected sites. This objective can be achieved by using uniform established procedures for sample collection, laboratory analysis, specific QA/QC procedures, and consistent reporting practices. The ability to meet this objective will be in part accomplished by adherence to the guidelines in this document.

The following sampling protocols must be followed when collecting, holding, and transporting PFAS samples for analysis using ***EPA Methods 1633 or 537 Modified (537M)***. The sample locations and sampling frequency are specified for all permittees in the accompanying letter. Samples shall be collected at the final outfall (s) and these locations are designated in the individual NPDES permits, the raw sewage influent line, and biosolids or sewage sludge area as specified.

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PFAS SAMPLING PROTOCOL

The potential presence of per- and polyfluoroalkyl substances (PFAS) in common consumer products, water used for decontamination, the equipment used to collect environmental samples from various media (soil, groundwater, surface water, sediment, and drinking water), and the use of extremely low reporting limits, requires special handling and care when collecting samples for PFAS analysis.

The instructions provided in this document serve as a guideline to avoid PFAS contamination and cross contamination during sampling events. It also discusses the analytical methods for PFAS sampling in municipal wastewater.

A. SAMPLE COLLECTION (Aqueous Samples - Influent and Final effluent)

1. General Sampling Guidelines

All planned sampling events should identify and address potential sources of contamination and cross-contamination prior to the initiation of each event.

- a. PFAS contamination during sampling can occur from several common sources, such as personal protection equipment (PPE), clothing, sun protection products, hand sanitizers, personal care products, food packaging, and sampling equipment.

Waterproof, water-repellant, or dirt and/or stain-repellent clothing should be avoided when collecting samples because these items probably contain PFAS. Tyvek® suits and clothing that contain Tyvek®, hard hats, and safety glasses may also contain PFAS and should, therefore, be evaluated before usage.

- b. Before sampling, field sampling staff may come into contact with materials and fabrics treated with PFAS, such as carpets and car interiors. Sampling staff should be cognizant that these materials may contain PFAS, and care must be taken to not contaminate sampling containers or sampling equipment.
- c. The sample collector must properly wash their hands before sampling and wear powder-free nitrile gloves while filling and sealing the sample bottles. This will aid in minimizing accidental contamination of the samples.
- d. Sample containers and equipment used for sampling should not be stored on or come into contact with materials suspected to contain PFAS.
- e. The sample container must be kept sealed and only opened during sample collection. The sampling container cap or lid should never be placed on the ground or on any other surface unless it is PFAS-free.

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- f. Samples do not need to be collected headspace free, unless specified. Collect separate samples for the matrix spike (MS) and matrix spike duplicate (MSD), if MS and MSD are required or requested.
- g. When collecting and handling aqueous samples, do not insert or let tubing or any materials inside the sample bottle. Dust and fibers must be kept out of sample bottles.
- h. After the samples have been collected and capped, they should be placed in individual polyethylene bags such as zip-lock bags and placed in a cooler that contains only regular ice. The ice and chain of custody (COC) sheets should always be bagged in (i.e., Ziploc®) bags.
- i. If you are collecting samples for other parameters at the same time, first collect the samples for PFAS and place these samples in a separate cooler. No other samples should be placed in the same cooler.
- j. The following procedures should be used to reduce the likelihood of sample contamination:
 - i. Wear clean, powder-free gloves.
 - ii. Do not touch the inside of the sample container/lid.
 - iii. Do not set the lid down.
 - iv. Minimize the time that the sample container is open.
 - v. Do not allow rainwater to drip into the sample container.
 - vi. Care should be taken to keep the sample containers dust-free prior to sample collection.
 - vii. Do not use sharpie regular tip markers as they may contain PFAS.
 - viii. Do not use waterproof field notebooks/paper in the sample staging area.
 - ix. Do not use any containers or sampling equipment that contains any known fluoropolymers.
 - x. Do not use clothing that has been washed with fabric softener, or washed with water, dirt, and/or stain-resistant chemicals. Do not use clothing chemically treated for insect resistance and ultraviolet protection.

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2. **Sample Collection Containers**

Grab samples should be collected in high-density polyethylene (HDPE) or polypropylene bottles with an unlined polypropylene or HDPE screw cap. These collection containers should be supplied by the testing laboratory. The containers should be capped, and the caps should not be removed until sample collection. All sampling equipment containing fluoropolymers must not be used for sampling or come in contact with the sample or container. These fluoropolymers include the following:

- ❖ Polyvinylidene fluoride (PVDF)
- ❖ Polychlorotrifluoroethylene (PCTFE)
- ❖ Polytetrafluoroethylene (PTFE)
- ❖ Ethylene-tetrafluoro-ethylene (ETFE)
- ❖ Fluorinated ethylene propylene (FEP)
- ❖ Polytetrafluoroethylene (PTFE)

The above fluoropolymers may go by the following trade names as well:

- ❖ Teflon®
- ❖ Hostaflon®
- ❖ Kynar®
- ❖ Tefzel®
- ❖ Neoflon®

PTFE (Teflon®) containers and contact surfaces with PTFE must be avoided to prevent contamination.

B. SAMPLE COLLECTION FOR DIFFERENT ANALYSIS METHODS

1. **Samples being analyzed by EPA Method 1633.**

- a. Aqueous samples are collected as grab samples. Collect 500 mL of sample (other than leachates) in an HDPE bottle. Do not fill the bottle past the shoulder, to allow room for expansion during frozen storage. Collect at least two aliquots of all aqueous samples to allow sufficient volume for the determination of percent solids and for pre-screening analysis.

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- b. Because the target analytes are known to bind to the interior surface of the sample container, the entire aqueous sample that is collected must be prepared and analyzed and subsampling must be avoided.
- c. When collecting leachate samples from landfills collect 100 mL of sample. Collect two 100-mL leachate sample aliquots in a similar manner as described in Section A.4.a, above, using appropriately sized containers.
- d. Maintain all aqueous samples protected from light at 0 - 6 °C from the time of collection until shipped to the laboratory. Samples must be shipped as soon as practical with sufficient regular ice to maintain the sample temperature below 6 °C during transport and be received by the laboratory within 48 hours of collection. The laboratory must confirm that the sample temperature is 0 - 6 °C upon receipt. Once received by the laboratory, the samples must be stored at ≤ -20 °C until sample preparation.

2. Samples being analyzed by EPA Method 537 Modified (537M)

- a. Aqueous samples shall be collected as a “Composite-Grab” for each sampling location using sampling containers provided by the laboratory.
- b. Collect 500ml composite samples by combining individual aliquots collected as grab samples at regular intervals, in 250ml HDPE bottles fitted with a polyethylene screw-cap lid. Do not fill the sample past the neck of the bottle.
- c. Maintain aqueous samples at a temperature of $\leq 6.0^{\circ}$ C from the time of collection till shipped to the laboratory. Ensure samples are shipped quickly enough and the cooler is filled with sufficient regular ice to keep the samples chilled during transport and shipment.

C. FIELD REAGENT BLANKS (FRB) COLLECTION

1. FRB must be collected along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. The testing laboratory will supply the reagent water, preservative, and collection container. At the laboratory, fill the field blank sample bottle with reagent water, seal, and ship to the sampling site along with the sample bottles.

For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the preserved reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAS were not introduced into the sample during sample collection/handling.

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2. The reagent water used for the FRBs must be initially analyzed for method analytes as a Laboratory Reagent Blank (LRB) (using the same lot of sample bottles as the field samples) and must meet the LRB criteria specified by the method QA/QC prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water or sample bottles rather than contamination during sampling.

D. SAMPLING OF BIOSOLIDS OR SEWAGE SLUDGE

1. Biosolids or sewage sludge contain both liquid and solid fractions. Therefore, the samples should be collected with the highest solids content possible. A uniform biosolids or sewage sludge sample may require the use of diapers or scoops that may be used for sampling. The equipment should be decontaminated prior to first use and between each sample collected to avoid cross-contamination.

Sampling equipment that will come into direct contact with the biosolids/sewage sludge must be PFAS-free prior to use. Do not use sampling equipment that contains any known fluoropolymers such as Teflon. Disposable sampling equipment that is certified to be PFAS-free is recommended. However, if this is not an option, stainless steel trowels or scoops as well as high-density polyethylene (HDPE) can be used to collect the samples.

To ensure comparability a grab sample consisting of equally sized samples should be collected from the same location. Laboratory analytical protocols require a minimum sample size to ensure analytical accuracy and meet the detection limits required for the project. Laboratories should be consulted in advance of any actual sampling to determine the minimum size sample required for the monitoring project.

Biosolids/sludge samples must be collected in PFAS-free containers with approved caps. These containers should be supplied by the testing laboratory.

2. All biosolids/sewage sludge samples including those with low solids content should be analyzed as solids and reported on a dry weight basis. This dry weight basis reporting requirement should be specified on the chain of custody or sample shipment form sent to the analytical laboratory.
3. The samples should be collected from a location that will yield a representative sample of the biosolids or sewage sludge generated by the WWTP.
4. Samples should be collected after any treatment processes and prior to leaving the WWTP for disposal. If liquids are present, a representative whole sample aliquot that includes both liquid and solid fractions should be collected. However, samples with the least amount of liquid are preferred.

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E. SAMPLE PREPARATION, HOLDING, STORAGE, AND TRANSPORT

Samples should be delivered to the laboratory for analysis as soon as possible following sample collection. It is recommended that this be done on the same day that the samples are taken.

| Matrix | Preservation | Holding Time |
|---------|-------------------------------------|--|
| Aqueous | Thermal: ≤ 6.0° C Chemical: None | Collection to Prep: 14 days Prep to Analysis: 28 days |
| Solid | Thermal: ≤ 6.0° C Chemical: None | Collection to Prep: 14 days Prep to Analysis: 28 days |

F. SAMPLE CUSTODY DOCUMENTATION - CHAIN-OF-CUSTODY FORMS

There must be documentation ensuring that proper sample handling has occurred throughout sampling, sample holding and transport to the laboratory. This documentation is a part of the custody record, which provides a mechanism for tracking samples through sample collection, processing, and analysis. Custody records document the “chain of custody”; the date and person responsible for the various sample handling steps associated with each sample. Chain of custody records also provide a documented trail of the handling of each sample to ensure regulatory requirements have been met. Precautions should be taken when labelling samples and documenting field activities to prevent contamination including the following:

1. Regular/thick size markers (E.G. Sharpie®) should not be used because they may contain PFAS. However, Fine and Ultra-Fine point Sharpie® markers are acceptable to label the empty sample bottle while in the staging area, provided the lid is on the sample bottle and gloves are changed following sample bottle labeling.
2. Ballpoint pens may be used to write on sample labels or preprinted labels from the laboratory may be used.

G. SAMPLE AND EXTRACT HOLDING TIMES

1. Method 537M under DoD QSM version 5.4, Appendix B Table B 15

- a. Aqueous samples should be extracted as soon as possible but must be extracted within 14 days of collection. Extracts must be stored at room temperature and analyzed within 28 days after the extraction.

2. Method 1633

- a. Aqueous samples (including leachates) should be analyzed as soon as possible; however, samples may be held in the laboratory for up to 90 days from collection, when stored at ≤ -20 °C and protected from the light. When stored at 0 - 6 °C and protected from the light, aqueous samples may be held for up to 28 days, with the

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caveat that issues were observed with certain perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids after 7 days. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.

- b. Biosolids samples may be held for up to 90 days if stored by the laboratory in the dark at 0 - 6 °C or at -20 °C. Because microbiological activity in biosolids samples at 0 - 6 °C may lead to the production of gases which may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, EPA recommends that samples be frozen if they need to be stored for more than a few days before extraction.

H. QUALITY ASSURANCE AND QUALITY CONTROL (QA/QC)

1. Method 537.1 and modifications under DoD QSM

All laboratory analytical quality assurance controls shall follow the guidelines and meet the requirements found in Table B.15 of the Department of Defense (DoD) and Department of Energy (DOE)'s *Consolidated Quality Systems Manual (QSM) for Environmental Laboratories Rev 5.3*.

2. Method 1633

All laboratory analytical quality assurance controls shall follow the guidelines and meet the requirements found in Method 1633 Section 9.0 Quality Control. A summary of all QA/QC can be found in Table 7 Summary of Quality Control found in the method.

I. FIELD QUALITY CONTROL

1. Field Blank

Field Blank/Field Reagent Blank: A sample of PFAS-free water is poured into the container in the field and shipped to the laboratory with field samples. Due to the prevalence of PFAS in a wide range of materials, there is the possibility of sample contamination in the field during the sampling, transport, and storage of samples. The purpose of the field reagent blank or field blank is to determine if the sampling activity caused any PFAS cross-contamination of the samples. This is required to verify that the sampling environment did not cause any contamination of the samples. Contact your laboratory to see if the field blank/reagent blank is required for your sampling project.

2. Field Duplicate

Field duplicates are replicate samples collected in the field and submitted to the laboratory as two distinct samples. Field duplicates are used to verify the precision of field and laboratory activities. The Field Duplicate (FD) is a sample collected from a sample location at the same time and under identical circumstances as the field sample and treated the same throughout field and laboratory procedures. These samples are optional.

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3. Equipment Rinsate Blanks

Equipment rinsate blanks are utilized to assess analyte contamination of the sampling apparatus used for the collection of samples. These blanks consist of PFAS-free water that is used to rinse the sampling equipment and collected after the sampling equipment has been thoroughly cleaned or decontaminated prior to sampling. Rinsate blanks must be handled in the same manner as the samples. Laboratory water and sample bottles used in the collection of rinsate blanks must be supplied by the laboratory which will be performing the analysis. The laboratories must certify that the bottles and water are PFAS-free. Check with the MDE to determine if you will be required to collect equipment rinsate blanks.

J. DEFINITIONS:

1. **BIOSOLIDS** - The semi-solid residual material created in a Wastewater Treatment Plant (WWTP) that is land applied in accordance with Federal and State regulations. This is treated sewage sludge that meets the EPA pollutant and pathogen requirements for land application and surface disposal.
2. **FIELD REAGENT BLANK (FRB)** – An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.
3. **HOLDING TIMES:** The maximum allowable time that may elapse from the time of sample collection to the time of sample preparation or analysis, or from preparation to analysis, as appropriate.
4. **INFLUENT-** The untreated wastewater or raw sewage coming into a wastewater treatment plant.
5. **MATRIX SPIKE (MS)** – A preserved field sample to which known quantities of the method analytes are added in the laboratory. The MS is processed and analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate sample extraction and the measured values in the MS corrected for background concentrations.
6. **MATRIX SPIKE DUPLICATE (MSD)** – A duplicate of the Field Sample used to prepare the MS. The MSD is fortified, extracted, and analyzed identically to the MS. The MSD can be used instead of the Field Duplicate to assess method precision when the occurrence of method analytes is low.
7. **MINIMUM REPORTING LEVEL (MRL)** – The minimum concentration that can be reported as a quantitated value for a method analyte in a sample following analysis.

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This defined concentration can be no lower than the concentration of the lowest calibration standard for that analyte and can only be used if acceptable QC criteria for this standard are met. The MRL is the lowest analyte concentration that meets Data Quality Objectives required by the method and or regulatory agency.

8. SEWAGE SLUDGE- The residual semi-solid material that is produced as a by-product during sewage treatment of industrial or municipal wastewater at WWTPs.

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QUICK FIELD GUIDE

| Clothing / Gear, Personal Care Products (PCP) & Personal Protective Equipment (PPE) | |
|--|--|
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ➤ Powder-free nitrile gloves ➤ Synthetic or 100% cotton clothing washed without fabric softeners. ➤ Clothing, boots, rain gear or PPE made of or with: Polyurethane Polyvinyl chloride (PVC) Wax coated fabrics Rubber / Neoprene Uncoated Tyvek ➤ Approved personal care/hygiene products or sun protection or insect repellent products applied in the staging area with hands thoroughly washed immediately after. | <ul style="list-style-type: none"> ➤ PCPs applied in the sampling area. ➤ Clothing that is waterproof or water-resistant (e.g. Gore-Tax, Scotchgard & RUCO) or treated with insect/water/dirt/stain-resistant chemicals. ➤ Clothing that is new or unwashed or washed recently with fabric softeners or fabric protectors ➤ Sun protection or insect repellent products applied in the sampling area. ➤ Hand sanitizer products ➤ Food packaging ➤ Latex gloves |
| Sampling Equipment & Items Used During Sampling Event | |
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ➤ Low-density polyethylene (LDPE) tubing. ➤ Equipment or items made from high-density polyethylene (HDPE), polypropylene, silicone, PVC, or stainless steel. ➤ Secure containers using natural rubber, nylon, or uncoated metal springs. | <ul style="list-style-type: none"> ➤ Items known or suspected to contain fluoropolymers, including: PFTE (Teflon/Hostaflon), PVDF (Kynar), PCTFE (Neoflon), ETFE (Tefzel) & FEP (Teflon FEP/Hostaflon FEP). ➤ Avoid PFTE containers and contact surfaces. |
| Sampling Storage & Preservation | |
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ➤ Glass jars for dry or solid samples, provided the laboratory can extract the adsorbed PFAS as part of sample preparation. ➤ Bottles/containers made of HDPE or polypropylene with unlined polypropylene or HDPE screwcaps. ➤ Regular wet ice | <ul style="list-style-type: none"> ➤ Glass jars for aqueous sample storage or when the laboratory may not be able to extract adsorbed PFAS during sample preparation. ➤ Aluminum foil known to be or potentially coated with PFAS. ➤ Chemical or blue ice ➤ LDPE bottles |

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| | |
|--|---|
| <ul style="list-style-type: none"> ☞ Thin HDPE sheeting ☞ LDPE resealable storage bags (e.g. Ziploc) that will not be in contact with the sample. | <ul style="list-style-type: none"> ☞ Polytetrafluoroethylene (PTFE)-lined bottles or caps ☞ Do not use any items or materials that are suspected or known to contain PFAS |
| Field Documentation | |
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ☞ Non-recycled & non-waterproof loose paper. ☞ Field clipboards made from aluminum, polypropylene or known PFAS-free materials. ☞ Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie markers. | <ul style="list-style-type: none"> ☞ Clipboards made with non PFAS-free materials. ☞ Notebooks, loose paper, or adhesive paper treated or coated with PFAS. |
| Decontamination | |
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ☞ Alconox, Liquinox, or Citranox ☞ PFAS-free deionized water (laboratory provided or commercially purchased) ☞ Cotton cloth or untreated paper towel | <ul style="list-style-type: none"> ☞ Decon 90 ☞ Paper towels treated with PFAS or not confirmed to be PFAS-free |
| Food & Beverages | |
| Allowable | Prohibited |
| <ul style="list-style-type: none"> ☞ Bottled water and hydration drinks brought into the staging area and consumed after removing PPE | <ul style="list-style-type: none"> ☞ Consumption of food or snacks in the staging or sampling area. ☞ Consumption of water or beverages in the sampling area. |