

**Oklahoma Department of Environmental Quality
Protocols for PFAS Sampling
Standard Operating Procedures**

Soil and Sediment Sampling SOP

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TABLE OF CONTENTS

1.0 GENERAL	4
2.0 FIELD CLOTHING AND PPE	4
3.0 EQUIPMENT	4
4.0 SAMPLING TECHNIQUES & COLLECTION METHODS	5
4.1 Surface Sampling	5
4.2 Subsurface Sampling – Hand Augers and Shovels	5
4.3 Subsurface – Direct Push	6
4.4 General Soil Sampling Guidelines	6
5.0 DECONTAMINATION	7
6.0 QUALITY ASSURANCE / QUALITY CONTROL	8
7.0 DOCUMENTATION	8
8.0 SHIPMENT	9

Table:

DEQ PFAS Sampling Quick Reference Field Guide

1.0 General

The objective of this protocol is to give general guidelines for the collection of soil samples for PFAS analysis. Multiple soil samples may need to be collected from the sampling site for composite, mix, or homogenized samples. Representative upgradient and downgradient PFAS samples should be collected before sampling the site to determine background PFAS concentrations. This guidance is based on the DEQ's research and USEPA Draft Method 1633 (expected to be finalized in 2022). The PFAS General Sampling Guidance should be referred to for what to include in a QAPP for PFAS sampling, information on cross-contamination, additional information on field clothing, and many other details.

2.0 Field Clothing and PPE

Field clothing and other personal protective equipment (PPE) typically may consist of PFAS-containing materials, especially those advertised as water-resistant, water repellent, or stain-resistant. Refer to Tables 1-4 and 8 in the Quick Reference Field Guide for general guidance regarding field clothing and PPE.

Sunscreen and biological protection also require screening, and recommendations are provided in Tables 5 and 7 in the Quick Reference Field Guide.

For guidance on specific brands and product names, refer to Table 6 in the Quick Reference Field Guide.

While PFAS-containing apparel and PPE should be avoided, the safety of samplers should never be compromised. Any deviation from the DEQ's guidance should be recorded in the field notes.

3.0 Equipment

All equipment used during sample collection should be assessed for the presence of PFAS to eliminate or reduce the probability of cross-contamination. Refer to Table 10 in the Quick Reference Guide at the end of this document for details on sampling containers that are and are not allowed. For soil sampling stainless steel, high-density polyethylene (HDPE), polypropylene, and silicone materials are recommended for collection. The standard equipment used in this process includes stainless-steel spoons and bowls, steel hand augers or shovels without any coatings, and direct push samplers with liners that are PFAS-free (such as acetate liners). Refer to Table 11 in the Quick Reference Guide at the end of this document for a list of common materials which are allowed or prohibited. Food packaging can contain PFAS, so food packaging and products should be kept in a designated eating area as noted in Table 9 of the Quick Reference Guide at the end of this document.

4.0 Sampling Techniques & Collection Methods

The following section provides guidance for soil sample collection for PFAS analysis. DEQ approves multiple soil sampling techniques. The selected sampling technique may vary depending on what is most suitable for the sampling entity, selected laboratory, site characterization, and release type. Soil sampling depth intervals should be considered prior to a sampling event, as PFAS concentrations are influenced by downward leaching during precipitation or irrigation events. Research indicates concentrations of PFAS are generally higher for soil samples collected just above the water table (capillary zone) due to air-water interfaces, than at other soil sampling intervals. The desired depth of sampling will determine which of the following sampling techniques are appropriate. These sampling techniques should be used in conjunction with the general guidance for soil sampling based on the DEQ's research and USEPA Draft Method 1633. Sample preparations should be outlined in a site-specific sampling plan, such as a Sampling Analysis Plan (SAP).

The sampling techniques outlined below are operating procedures for soil collection and sampling based on USEPA Draft Method 1633, USEPA Region 4 Laboratory Services and Applied Science Division (LSASDPROC-300-R4), and the DEQ's research.

4.1 Surface Sampling

- If vegetative turf, gravel, or concrete are present at or near the surface sampling site, these should be removed using a pre-cleaned PFAS-free trowel or shovel before sampling. The vegetative turf should be set aside to be replaced at the conclusion of the sampling.
- Surface soil samples (typically 0 to 6 inches below the surface) should be collected using a pre-cleaned PFAS-free stainless steel spoon. The depth of the measurement begins at the top of the soil horizon, immediately below the removed materials.

4.2 Subsurface Sampling – Hand Augers and Shovels

- For shallow subsurface soil samples (typically 6 to 36 inches below the surface), a pre-cleaned PFAS-free hand auger or shovel may be used to dig a hole and collect the sample. When the desired subsurface depth is reached, a pre-cleaned hand auger bucket or shovel shall replace the one used to reach the desired sampling depth to obtain the actual sample.
- When power augers are used to advance the borehole to the desired depth for sampling, care should be taken to ensure that exhaust fumes, gasoline, or oil will not contaminate the borehole or area in the immediate vicinity.
- The auger bucket could scrape the sides of the auger hole; therefore, it is recommended the top several inches of soil in the auger bucket be discarded before placing the bucket contents into a stainless-steel or other PFAS-free

bowl for mixing. The soil should be deposited directly into a stainless-steel bowl for mixing, and the material should be mixed thoroughly by rolling the material into the middle of the bowl until it is homogenized. At this point, the sample in the stainless-steel bowl can be placed into the laboratory-provided container(s).

4.3 Subsurface – Direct Push

- The depth of the measurement for the sample begins at the top of the soil horizon, immediately below the removed materials.
- If gravel or concrete is present at or near the surface sampling site, these should be removed prior to sampling. The turfgrass is typically not removed before sampling when using direct push devices.
- Direct push sampling methods are utilized to collect shallow and deep subsurface soil samples that involve collecting and retrieving a core soil sample within a thin-walled liner.
- Coring mechanisms should consist of a stainless-steel core barrel with a clear plastic liner (use an acetate or another PFAS-free liner) into which the soil core will enter. If a core sampler includes an HDPE sleeve inserted in the core barrel to retain the sample, the sampler must ensure that materials that come in contact with the sampled media do not contain water-resistant coatings that contain PFAS.
- Once the core barrel is retrieved at the surface, this liner should be removed, placed on the cutting board, and opened using a liner cutting device. The soil sample should then be visually inspected with observations recorded in the site field book. The core should be cut open to reveal a "clean" face for sampling. This process avoids the possibility of any cross-contamination that may have been introduced as the soil core entered the liner.

The general sampling guidelines below are presented to supplement and further clarify the collection methods listed above.

4.4 General Soil Sampling Guidelines

- Studies have shown there is a loss of PFAS due to adsorption to surfaces; as a result, samples should be minimally handled and directly placed in the sample container as soon as possible.
- To minimize PFAS cross-contamination, grab samples are preferable. If chosen to composite, mix or homogenize samples, it is recommended that this be done at the laboratory to provide a representative subsample to be analyzed. At this time, the State Environmental Laboratory is unable to perform homogenization of samples. When the sampling technician conducts homogenization in the field, USEPA recommends that additional grab samples be provided to determine the variability and impacts on the PFAS concentrations of the mixed samples.

- To minimize PFAS cross-contamination, the DEQ recommends the trace level sampling technique which involves two field personnel to collect the samples. When the field investigator is ready to fill the sample container, a designated sampler will apply new gloves, while the field investigator, also with new gloves, will open the sample container packaging. The designated sampler removes the sample container(s) from the packaging or Whirl-Pak® while keeping it closed until ready to fill, at which time the designated sampler will remove the cap and hold the container until an ample sample is obtained. The field investigator fills the sample container. The designated sampler holding the sample container should touch nothing else during the collection process. The field investigator will operate the PFAS-free sampling equipment and assist with the packaging and labeling of the sample container.
 - Solid samples should be collected in laboratory-provided containers that are wide-mouthed, hold approximately 500 milliliters (mL), made of HDPE, with linerless polypropylene or HDPE caps. The container should be filled three-quarters (3/4) full.
 - Solid samples must be maintained between +4°C and - 2°C, protected from light in the HDPE containers from the time the samples are collected until delivery to the laboratory.
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If further information regarding each sampling technique is desired before sampling, please reference the respective USEPA method documents mentioned.

5.0 Decontamination

Refer to Table 13 in the Quick Reference Field Guide at the end of this document for guidance on decontamination methods and materials which are allowed or prohibited while sampling. The following general decontamination principles for PFAS sampling should be followed:

- Sampling equipment must be decontaminated after sampling at each location or at the end of the workday.
- Wash hands with PFAS-free water and put on new nitrile gloves before decontamination.
- Decontaminate equipment with a triple rinse of verified PFAS-free water and remove particulates with a polyethylene or PVC brush.
- PFAS-free detergents such as Alconox®, Liquinox®, or Citranox® may be used.
- Dry equipment with a cotton cloth, untreated paper towels, or place decontaminated equipment on a PFAS-free surface to air-dry.

- If decontamination of the drill rig equipment consists of steam washing with PFAS-free water, care should be taken to contain the steam and water. PFAS may aerosolize during decontamination procedures and deposit on the site surface, creating a contamination risk.

6.0 Quality Assurance / Quality Control

The DEQ recommends following the Quality Assurance/Quality Control (QA/QC) Guidelines outlined in section 5.0 of the General PFAS Sampling document to ensure project-specific Quality Assurance Project Plan (QAPP), Standard Operating Procedures (SOP), and Sampling Analysis Plan (SAP) consistency between sampling events. Additionally, the following guidelines should be followed for soil-specific sampling events.

- Collect a rinsate/equipment blank at the rate of 1 per day per sampling team to ensure the sampling has not introduced PFAS cross-contamination to the sampling results. This process should be conducted on each piece of equipment. (Required)
- Laboratory sources of water used for equipment decontamination and blank sample collection should be certified PFAS-free or addressed for background concentrations of PFAS. (Required)
- Collect field quality control samples such as duplicates at the rate of 1 per 10 samples, trip blanks at the rate of 1 per cooler per sampling team, and field blanks at the rate of 1 per day per sampling team to assist in evaluating soil sampling and handling activities at the investigation site. (Required)
- If possible, a control sample/background sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. The soil sample should be collected as close to the sampled area and from the same soil type to document that low-level contaminants were not introduced by the sampling tools. (Required)

7.0 Documentation

Keep a sampling log during the sampling event. In the sample log, record the following:

- Sample point location
- Owner's Contact Information (if applicable)
- Sample borehole
- Depth of sample
- Sampling equipment
- Duplicate sample(s)
- Visual description of samples
- Use of any unapproved PPE
- Other sampling specific (applicable) observations

Ensure documentation materials are PFAS-free; refer to Table 12 in the Quick Reference Field Guide. Pre-printed labels for sample containers are preferred.

8.0 Shipment

The following is recommended for sample shipment. Information is also provided in the Quick Reference Field Guide Table 14.

- Use regular ice, double-bagged, in place of chemical (blue) ice and maintain the temperature between +4°C and -2°C in a cooler.
- Check the cooler periodically to ensure samples are well iced and at the proper temperature.
- The cooler should be taped closed with a custody seal.
- Double bag Chain of Custody and other forms and tape to the inside of the cooler lid.
- Ship within 48 hours or per the holding time determined by the laboratory or the selected laboratory analysis method.

DEQ PFAS Sampling Quick Reference Field Guide

Table 1: Clothing ¹		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Well laundered clothing (recommended six times prior to sampling) • 100% cotton (preferred) • Synthetic fabrics • Polyvinyl Chloride (PVC) • Polyurethane • Uncoated Tyvek® clothing • Wax-coated fabrics. • Rubber/Neoprene 	<ul style="list-style-type: none"> • New/unwashed clothing • Clothing applied/washed with fabric softeners, fabric protectors including ultraviolet (UV) protection, water, dirt or stain-resistant chemicals, or insect-resistant chemicals • Clothing containing Tyvek® • Flame resistant (FR) clothing • Clothing made of Gore-Tex or other known PFAS containing materials. 	<ul style="list-style-type: none"> • Tyvek® suits, clothing that contains Tyvek®, or coated Tyvek®

¹Clothing should be kept dust and fiber free.

Table 2: Boots		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Polyurethane boots • PVC boots • PFAS-free boot covers 	<ul style="list-style-type: none"> • Gore-Tex® boots • Boots made from water-resistant synthetics 	

Table 3: Gloves		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Powderless nitrile gloves* 	<ul style="list-style-type: none"> • Gore-Tex gloves • Any glove made with PFAS-containing materials. 	<ul style="list-style-type: none"> • Latex gloves • Water and dirt-resistant leather gloves • Any special gloves required by a Health and Safety Plan (HASP).

¹Samplers must wash their hands with PFAS-free water before putting on any gloves.

Table 4: PPE ¹		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Hard hats made of HDPE • Hard hat covers/liners (i.e. Head Gaiters) made of cotton or other natural fabric • Safety glasses made of HDPE • Life jackets made of polyethylene foam and nylon shell fabric • Waders made of Neoprene or other PFAS-free material 	<ul style="list-style-type: none"> • Waders made of Gore-Tex or other known PFAS containing materials 	<ul style="list-style-type: none"> • Hard hats or safety glasses not made of HDPE

¹PPE should be kept dust and fiber free.

Table 5: Sun Protection		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Approved Sunscreens (See Table 6) 	<ul style="list-style-type: none"> • No unauthorized sunscreen 	

Table 6: Allowed/Approved Sunscreens ¹
• Banana Boat® for Men Triple Defense Continuous Spray Sunscreen SPF 30
• Banana Boat® Sport Performance Coolzone Broad Spectrum SPF 30
• Banana Boat® Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
• Banana Boat® Sport Performance Sunscreen Stick SPF 50
• Coppertone® Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
• Coppertone® Sport High-Performance AccuSpray Sunscreen SPF 30
• Coppertone® Sunscreen Stick Kids SPF 55
• L'Oréal® Silky Sheer Face Lotion 50
• Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 50
• Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30
• Meijer® Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
• Meijer® Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70
• Neutrogena® Beach Defense Water+Sun Barrier Lotion SPF 70
• Neutrogena® Beach Defense Water+Sun Barrier Spray Broad Spectrum SPF 30
• Neutrogena® Pure & Free Baby Sunscreen Broad Spectrum SPF 60+
• Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30

¹Baby sunscreens that are "free" or "natural" are not guaranteed PFAS-free and need additional research.

Table 7: Insect Protection ¹		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • OFF® Deep Woods • Sawyer® Permethrin 	<ul style="list-style-type: none"> • No unauthorized insect protection 	

¹Approved sunscreens and insect repellents should not be applied near the sample collection area. Hands should be well washed after application or handling of these products, and afterwards; an uncontaminated clean/new pair of powderless nitrile gloves should be worn.

Table 8: Prohibited Water Repellant Field Clothing and PPE Brand and Product Names	
• Ultra Release Teflon®	• Release Teflon®
• Repel Teflon® Fabric Protector	• High-Performance Release Teflon®
• High-Performance Repel Teflon® Fabric Protector	• Advanced Dual Action Teflon® Fabric Protector
• NK Guard® S Series	• GreenShield®
• Tri-Effects Teflon® Fabric Protector	• Lurotex Protector RL ECO®
• Oleophobol CP®	• Repellan KFC®
• Rucostar® EEE6	• Unidyne™
• Bionic Finish®	• RUCO-GUARD®
• RUCOSTAR®	• RUCO-COAT®
• RUCO-PROTECT®	• RUCOTEC®
• RUCO®	• Resist Spills™
• Resists Spills and Releases Stains™	• Scotchgard™ Fabric Protector
• GoreTex®	

Table 9: Food Containers		
Allowed	Not Allowed	Needs Additional Research
• Food packaging and products in a designated eating area set up for food and beverage consumption	• Food packaging and products in the staging or sampling areas	• Bringing foods rewrapped in PFAS-free materials

Table 10: Sampling Containers		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • HDPE also known as polyethylene high-density (PEHD) • Polypropylene • Stainless Steel • Unlined bottle caps • LDPE resealable bags (Ziplock) that will not come in contact with the sample media • 	<ul style="list-style-type: none"> • Polytetrafluoroethylene (PTFE) lined bottles or caps (i.e. Teflon® and Hostaflo®) • LDPE containers that will contact the sample media • Aluminum foil is not to be used due to the possibility of it being coated with PFAS. Utilize an alternative sample preparation and storage material. 	<ul style="list-style-type: none"> • Glass bottles and containers¹

¹Glass bottles or containers may be used if they are known to be PFAS-free; however, PFAS have been found to adsorb to glass, especially when the sample is in contact with the glass for an extended period of time (e.g., stored in a glass container). If the sample comes into direct contact with the glass for a short period of time (e.g., using a glass container to collect the sample, then transferring the sample to a non-glass sample bottle), the adsorption is minimal. Generally, glass bottles or containers should not be used for PFAS samples.

Table 11: Sampling Equipment		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • HDPE (also PEHD) • Polypropylene • Stainless Steel • Acetate • Silicone 	<ul style="list-style-type: none"> • Polytetrafluoroethylene (PTFE) • Polyvinylidene fluoride (PVDF) • Polychlorotrifluoroethylene (PCTFE) • Ethylene-tetrafluoroethylene (ETFE) • Low-density polyethylene (LDPE) which will contact the sample media 	<ul style="list-style-type: none"> • Glass equipment

Table 12: Field Materials		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> • Aluminum, polypropylene, or Masonite field clipboards • Rite in the Rain® notebooks • Loose paper (non-waterproof, non-recycled) • Ballpoint pens and pencils 	<ul style="list-style-type: none"> • Clipboards coated with PFAS-containing materials • Notebooks made with PFAS treated paper • PFAS treated loose paper • Post-It® Notes or other adhesive paper products • Sharpie® markers • Coated materials, including paper towels • Aluminum foil is not to be used due to the possibility of it being coated with PFAS. Utilize an alternative sample preparation and storage material. 	<ul style="list-style-type: none"> • Plastic clipboards, binders, or spiral hardcover notebooks • Waterproof field books • All markers not listed as allowable

Table 13: Decontamination Procedures		
Allowed	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> Alconox®, Liquinox®, or Citranox® Triple rinse with PFAS-free water Cotton cloth or untreated paper towels Polyethylene or PVC brush to remove particulates 	<ul style="list-style-type: none"> Decon 90® PFAS treated paper towels Reusing non-dedicated equipment without decontaminating 	<ul style="list-style-type: none"> Municipal water¹ Recycled or treated paper towels

¹Decontamination procedures should include a triple rinsing with PFAS-free water for equipment such as dippers, balers, spades, etc. Laboratory supplied PFAS-free deionized water is preferred for cleaning and decontamination. However, commercially available deionized water may be used for cleaning and decontamination if the water is verified to be PFAS-free. Municipal drinking water may be used for cleaning or decontamination if the water is known to be PFAS-free.

Table 14: Sample Shipment		
Allowed/Required	Not Allowed	Needs Additional Research
<ul style="list-style-type: none"> Coolers filled with regular ice Maintaining sample temperature between +4°C and -2°C Double-bagging of samples and ice using bag materials made of HDPE (preferred) or LDPE (if sample does not come in contact) Chain of Custody and other forms should be single bagged in LDPE (e.g. Ziploc®) storage bags and taped to the inside of the cooler lid. 	<ul style="list-style-type: none"> Aluminum foil is not to be used due to the possibility of it being coated with PFAS. Utilize an alternative sample preparation and storage material. Chemical (blue) ice packs 	<ul style="list-style-type: none"> Chemical (blue) ice packs that are verified PFAS free