

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

Fish Sampling SOP

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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 fish tissue samples for PFAS analysis. PFAS concentrations in fish can be several orders of magnitude higher than in ambient water; analysis in fish tissue often results in parts per billion (ppb) while ambient water measures in parts per trillion (ppt). Tissue analysis may be useful in understanding the bioaccumulation of PFAS, conditions in the ambient water, and concentrations of the substances in fish tissue that are consumed. This guidance is based on the DEQ's research and USEPA Draft Method 1633. 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 Apparel and PPE

Field clothing and other PPE 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 at the end of this document 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 at the end of this document.

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

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 fish sampling, a variety of equipment may be used depending on the project's goals. 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.

Additional information to consider when selecting sampling equipment is as follows:

- Studies have shown that cross-contamination during the collection and processing of fish samples before delivery to the analytical laboratory is unlikely to affect the results.
- Reasonable precautions should still be taken to avoid cross-contamination while selecting equipment for collecting, measuring, and filleting fish in the field.

4.0 Sampling Techniques and Collection Methods

The following section provides guidance for fish tissue sample collection for PFAS analysis. DEQ approves multiple fish tissue 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. For fish tissue sampling, the purpose of analysis (understanding PFAS accumulation, determining risk to anglers, etc.) will typically determine which sampling equipment and methods are the most suitable. Different fish tissues can have varying propensities to accumulate PFAS; therefore, it is crucial to communicate with the laboratory in advance to determine how the fish tissue sample should be prepared and sent to the laboratory. There are currently no USEPA methods that give comprehensive fish tissue guidance. In the absence of method-specific guidelines to follow, general guidance for fish tissue sampling based on the DEQ's research and USEPA Draft Method 1633, a recently published method slated for multi-laboratory validation by early 2022, is given below. Sample preparations should be outlined in a site-specific sampling plan, such as a Sampling Analysis Plan (SAP).

4.1 General Fish Tissue Sampling Guidelines

- Field sampling plans and protocols should explicitly state the samples to be collected and if any processing will be conducted in the field (e.g., filleting the fish)
- Keep a fish collection record form to track specimen collection (example shown at the end of this section). This, along with a Chain of Custody, should accompany the samples to the lab.
- Fish size (length and weight) should be recorded after collection and before placement into storage containers.
- Each specimen should be individually tagged through a jaw tag or a tag within/on the specimen holding container. Affix the tags so that they can be read without opening the container. For composite-style samples, a tag can be placed in or on the container with the specimens. Tag names or numbers should be recorded on the fish collection record form.
- Each specimen should be placed into its own container to avoid difficulties or cross-contamination.
- Do not fillet or clean the fish unless specifically asked to do so. USEPA Draft Method 1633 states, "fish may be cleaned, filleted, or processed in other ways in

the field, such that the laboratory may expect to receive whole fish, fish fillets, or other tissues for analysis.”

- As soon as data collection and recording is completed, fish should be kept cool at a temperature of at least 8° C. Fish should be frozen as soon as possible.

Below is an example of a fish collection form. The DEQ does not require or suggest this particular form be used but instead advises using some form to track specimen collection.

Fish Collection Record Form									
Project and Site Name: _____									
Collections Made By: _____									
Sampling Method: _____									
For Lab Use Only- Lab Entry No.	Collection or Tag No.	Species	Date Taken	Location	Age	Sex or Reproductive Condition	Length	Weight	Comments

4.2 General Guidelines for Selection of Fish Samples

- Ideally, a minimum of 10 fish, including samples from both sexes, should be captured for analysis.
- If assessing the human health risks is the project goal, then fish species and sizes typically consumed by anglers should be chosen.
- If assessing ecological risks is the project goal, then fish species and sizes normally consumed by piscivorous mammals and birds should be targeted.

- Consider selecting species that have been sampled in other Oklahoma waters to allow more accurate spatial comparisons.
 - Any migratory patterns of fish should be considered when using the data to assess the waterbody conditions in which the fish are captured.
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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 don new nitrile gloves before decontamination.
- Decontaminate equipment with a triple rinse of verified PFAS-free water. A Polyethylene or PVC brush to remove particulates and 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.

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 fish-specific sampling events.

- Laboratory sources of water used for equipment decontamination and blank sample collection should be produced as PFAS-free or addressed for background concentrations of PFAS. (Required)

7.0 Documentation

Keep a fish collection record form during the sampling event. In the form, record the following:

- Sample point location
- Sampling equipment
- Use of any unapproved PPE
- Each unique fish tag number
- Species identification
- Date Collected
- Sample location
- Fish size (length and weight)
- Fish sex
- 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 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 applicable forms and tape to the inside of the cooler lid. Include the appropriate Fish Collection Record forms.
- Ship within 48 hours or per the holding time determined by the laboratory or the selected laboratory analysis methods.

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 Hostaflon®) • 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