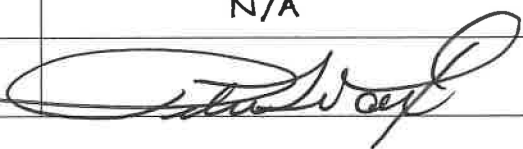




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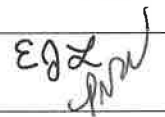
### AUTHORIZATIONS

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### QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Collection and Processing of Whole Fish Tissue Samples  
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Revision	Change Description	Date	Authorization
1	PFAS Considerations	2/25/2023	

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(Place to acknowledge peer reviewer)

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## **1.0 SCOPE AND APPLICABILITY**

This document presents the North Dakota Department of Environmental Quality, Division of Water Quality's (DWQ) Standard Operating Procedure (SOP) for collecting and processing of whole fish tissue samples. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers.

## **2.0 SUMMARY OF METHOD**

Fish spend their entire life in a waterbody which makes them an important indicator of water quality, especially toxic pollutants. Toxic pollutants, which may be present in the water column or the sediments at concentrations below our analytical detection limits, may be exhibited in fish tissue analysis due to bioaccumulation.

Composite whole fish samples are analyzed for major organic contaminants (i.e., PCBs and pesticides) and trace metals including mercury. Table 7.13.2 contains a complete list of the parameters analyzed. The data generated is used to assess the impacts and the extent of contamination in our lakes and streams. The data is also used to determine which waterbodies require additional sampling for the possible issuance of fish consumption advisories.

A composite sample of similarly sized and like species of fish are collected and ground whole. The composite is mixed well, and a 500 to 1000 ml sample is placed in a glass jar with Teflon lid. The sample is labeled and immediately frozen to await chemical analysis.

## **3.0 HEALTH AND SAFETY WARNING**

Field personnel should take appropriate precautions when operating electrofishing gear on, in, or around the water. All sampling crews should be equipped with personal protective equipment (PPE). This equipment would include non-breathable waders, rubber gloves, eye protection, etc. When operating a boat, the North Dakota's boating laws and rules shall be followed by all field personnel.

Field personnel should be aware that hazardous conditions potentially exist at every waterbody. If unfavorable conditions are present at the time of sampling, the sample visit is recommended to be rescheduled. If hazardous weather conditions arise during sampling, such as lightning or high winds, personnel should cease sampling and move to a safe location.

## **4.0 CAUTIONS**

The length of any one fish in the composite group should not exceed  $\pm 25$  percent of the average length of the entire composite group. The largest fish should be collected. Use latex gloves when sampling and processing samples. DO NOT freeze sample until processed in the lab. Samples can only stay on ice or freezer packs for a max of 48 hours.

## **5.0 INTERFERENCES**

Prior to processing (grinding) the first sample and after processing each composite sample, wash the grinder assembly, collection pan, cutting board, and knives with hot tap water, rinse with acetone and allow to air dry. This will prevent sample contamination between samples and provide accurate reliable data.

## **6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES**

All personnel collecting and processing whole fish tissue samples must read this SOP annually and acknowledge they have done so via a signature page (see Appendix B). New field personnel must also demonstrate successful performance of the method. The signature page will be signed by both trainee and trainer to confirm that training was successfully completed and that the new monitor is competent in carrying out this SOP. The signature page will be kept on-file at DWQ along with the official hard copy of this SOP.

## 7.0 EQUIPMENT AND SUPPLIES

### Field Equipment and Supplies

- \_\_\_\_\_ Copy of this SOP
- \_\_\_\_\_ Fish measuring board
- \_\_\_\_\_ Fish weigh scale
- \_\_\_\_\_ Plastic bags
- \_\_\_\_\_ Coolers with ice or frozen gel packs
- \_\_\_\_\_ Field data forms
- \_\_\_\_\_ Sample labels
- \_\_\_\_\_ Sample log forms
- \_\_\_\_\_ Waders (when shocking use pvc coated chest waders)
- \_\_\_\_\_ Raincoat
- \_\_\_\_\_ Rubber gloves
- \_\_\_\_\_ Pen
- \_\_\_\_\_ Fish collection gear (nets, electrofishing gear, etc.) if necessary
- \_\_\_\_\_ 5-gallon bucket
- \_\_\_\_\_ Generator (if electrofishing)

### Laboratory Equipment and Supplies

- \_\_\_\_\_ Knife(s)
- \_\_\_\_\_ Sharpening stone
- \_\_\_\_\_ Meat grinder (Fleetwood Model T 22 Chopper) with stainless steel feed pan, cylinder, worm gear, blades, and sieve plate
- \_\_\_\_\_ Stainless steel pan
- \_\_\_\_\_ Acetone (reagent grade)
- \_\_\_\_\_ Soap
- \_\_\_\_\_ Sample containers (Qorpak, EPA Clean, 8-oz. glass jars with Teflon-lined cap)
- \_\_\_\_\_ Sample labels
- \_\_\_\_\_ Sample ID/Custody Report Forms
- \_\_\_\_\_ Pen
- \_\_\_\_\_ Powderless Nitrile Gloves

## 8.0 FIELD PROCEDURE

Upon arrival to the sample site, establish which sampler is going to collect the whole fish sample.

1. A minimum of two composite samples are collected for analysis. One composite group should be represented by a large predator species (e.g., northern pike, walleye, largemouth bass) the other group should be represented by a bottom-feeding species (e.g., carp, white sucker, redhorse, catfish).
2. Fish will usually be collected in conjunction with the North Dakota Game and Fish Department's annual test netting operations. When collecting fish in conjunction with the Game and Fish, a special effort should be made to coordinate schedules to not jeopardize the quality of the fish collected for analysis. The following methods are commonly employed by the Game and Fish: trap netting, gill netting, and electrofishing. In general, any method of collection is acceptable providing the samples are fresh and in good condition.
3. Sort the fish collected by species and by size. Select five fish (three minimum) within each group for composite analysis. Each composite group should consist of fish of uniform size. As a guideline, the length of any one fish in the composite group should not exceed  $\pm 25$  percent of the average length of the entire composite group. The largest fish possible should be collected.
4. Fill out the fish tissue collection field data form (Figure 7.13.1), recording the species, sex (if possible, to determine), length, and weight.
5. Place a sample label on the plastic bag containing the composite fish sample (Figure 7.13.3).
6. Place the samples in a cooler on ice! Note: Fish may be kept refrigerated or on ice for up to 48 hours after collection. They must not be frozen until they are processed in the laboratory.



## 9.0 PFAS Considerations

When testing for PFAS extra precautions and steps must be taken to prevent cross contamination. It has not yet been documented that cross-contamination of fish tissue in the field setting has a significant impact on laboratory results, however staff should be aware of the potential PFAS sources and attempt to minimize potential effects. Michigan Department of Environmental Quality created a [quick reference guide](#) to illustrate the proper and improper materials for the field and laboratory. The MDEQ PFAS sampling quick reference field guide is attached.

## 10.0 LABORATORY PROCEDURE

1. Prior to processing (grinding) the first sample and after processing each composite sample, wash the grinder assembly, collection pan, cutting board, and knives with hot tap water, rinse with acetone and allow to air dry. When sampling for PFAS, ensure the tap water is PFAS free.
2. Wear powderless nitrile gloves when processing samples and change gloves between processing composite samples.
3. Cut up each fish into small pieces and pass through the grinder once.
4. Hand mix the composite sample until thoroughly homogenized, then pass through the grinder a second time.
5. Hand mix the sample a second time then fill a sample container with the sample (one pint of sample is equivalent to approximately 500 grams).
6. Label the sample container appropriately and fill out the Sample ID/Custody Report (7.13.2).
7. If the sample log form indicates a split sample be collected, fill a second sample container and label appropriately (Figure 7.13.3). Note: Fish tissue split samples should be identified with STORET number 389995.
8. Place the sample containers in the freezer prior to submitting the samples to the laboratory.
9. If another composite sample requires processing, repeat steps (1) through (7)

## **11.0 DATA AND RECORDS MANAGEMENT**

Fish data will be recorded on the field form 7.13.1 (Appendix A). Once personnel reach the office, data recorded on the field form are entered into the DWQ Sample Identification Database (SID). Field notes should be used to record any quality control activity performed, such as measurements taken by more than one sampler, or to record any sampling conditions that may have interfered with the data collected. Field forms and notes should be stored in the appropriate project folder at DWQ.

## **12.0 QUALITY ASSURANCE AND QUALITY CONTROL**

Quality assurance and quality control (QA/QC) procedures will be followed as explained above. Individual will have to follow the field and laboratory standard operating procedures to comply with the QA/QC for collecting and processing whole fish tissue samples.

## **13.0 REFERENCES**

National Rivers and Streams Assessment 2018/19: Field Operations Manual  
EPA-841-B-17-003a

### **Related DWQ SOPs**

7.14 Fish Skin on Fillet Tissue Sample Collection

7.15 Fish Tissue Plug Samples for Mercury Analysis

**APPENDIX A**  
Field Reporting Form

**Figure 7.13.1** Fish tissue collection field data form.

**Surface Water Sample Identification Code R (Tissue samples)**

Samples received without this sheet or without all bold sections fully completed will be rejected and not analyzed.

**Sample Collection/Billing Information**

Account #	Project Code:	Project Description:	
Customer (Name, Address, Phone):			
Date Collected:	Time Collected:	Matrix: Tissue	Site ID:
Site Description:			
Alternate ID:		Collected By:	
County Number:	County Name:		
Comment:			
Comment:			

**Field Information/Measurements**

Species Name:	Species Code:	Tissue Type:	Sample Size:
Comment:	Min. Length (cm):	Max. Length (cm):	Ave. Length (cm):
	Min. Weight (g):	Max. Weight (g):	Ave. Weight (g):

**Analysis Requested**

■ 13185) Mercury	■ 2434) Selenium		
■ 77) Base/Neut. Pest			
■ 78) Trace Metals			
■ 106) Acid Herbicides			
■ 107) PCBs			
■ 112) Urons			
■ 113) Carbamates			
■ 143) PAHs			

Figure 7.13.2 Fish sample custody form.

<b>Sample ID</b>	<b>Project Code</b>	<b>Project Description</b>
<b>Analysis: (DC Code) SW-Analyte Group</b>		
<b>Fish Species</b>	<b>Composite Size</b>	
	<b>Type of sample</b>	<b>Composite Weight</b>
	<b>Container:</b>	<b>Preservative</b>
<b>Date: _/_/_ Time: __ Depth:</b>		
<b>Sampler</b>		

	<b>Project Code</b>	<b>Project Description</b>
<b>389995</b>		
<b>Analysis: (DC Code) SW-Analyte Group</b>		
<b>Fish Species</b>	<b>Composite Size</b>	
	<b>Type of Sample</b>	<b>Composite Weight</b>
	<b>Container:</b>	<b>Preservative:</b>
<b>Date: _/_/_ Time: __ Depth:</b>		
<b>Sampler</b>		

**Figure 7.13.3** Fish flesh label, and fish flesh split label.

**APPENDIX B**  
SOP Acknowledgement and Training Form



# SOP Acknowledgement and Training Form

This SOP must be read, and this form signed annually. This form must be kept with the latest version of the SOP.

<b>Document Title:</b>	
<b>Document Revision Number:</b>	
<b>Document Revision Date:</b>	

Please sign below in accordance with the following statement:

"I have read and understand the above referenced document. I agree to perform the procedures described in this SOP in accordance with the document until such time that it is superseded by a more recent approved revision."

[illegible]

## SOP Acknowledgement and Training Form (con't)

Trainee: Sign below to acknowledge that training on this SOP was received, understood, and all questions/concerns were addressed by the trainer.

**Trainer:** Sign below to acknowledge that training on this SOP was completed for the individual listed and that training is competent to perform the procedures described within.

[illegible]