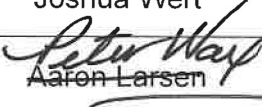





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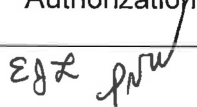
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QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Collection and Processing of Fish Tissue Plug Samples for Mercury Analysis
Type: Standard Operating Procedure 7.15
Version: 3.0
Date: 02/07/2020
Author: Joshua Wert

REVISION HISTORY

Revision	Change Description	Date	Authorization
1	Department address updated	06/08/2023	

ACKNOWLEDGEMENTS

(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 fish tissue plug samples for mercury analysis. 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.

Typical fish tissue collection methods require the fish to be sacrificed, whether it be a whole fish or a skin on fillet tissue sample. This can be problematic when there is a need to collect large trophy sized fish for contaminant analysis or when a large sample size is necessary for statistical analysis. The following describes an alternative method for the collection of fish tissue samples which uses a tissue plug instead of a skin on fillet. This method is advantageous in that it eliminates the need to kill the fish to obtain a fish tissue sample for mercury analysis. Secondly, skin on fillet sampling required homogenizing of samples through a grinder. Although the grinder is cleaned between samples, the risk of sample contamination is a concern. The plug method uses clean equipment and supplies each time a sample is collected, thus reducing the risk of sample contamination.

A plug tissue sample is collected by inserting a biopsy punch into a de-scaled meaty section of a live fish. After collection antibiotic salve is placed over the wound and the fish is released.

Fish tissue sampling is conducted in conjunction with the North Dakota Game and Fish Department's (NDGFD) spring and fall spawning operations. Fish tissue sampling is also conducted throughout the summer months in conjunction with the NDGFD's test netting operations on specified lakes.

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 should 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 largest and smallest fish within each group should not exceed the average length of the group by more than 25%.

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.

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
- _____ Sterile 20 mL glass scintillation vials
- _____ 8-millimeter disposable biopsy punch (Acuderm brand Acu-Punch or equivalent)
- _____ Laboratory pipette bulb
- _____ 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)

8.0 FIELD PROCEDURE

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

1. Collect up to five fish per species of similar size ranges. Size ranges should be visually obvious. As a general guideline, the largest and smallest fish within each group should not exceed the average length of the group by more the 25%.
2. Acceptable methods for fish collection include hoop net, electro-fishing, trap net, hook and line, or any method in which the fish sample will remain alive. However, methods in which the fish is sacrificed may also be used. These include rotenone, gill netting, or any other method which provides fresh fish in good condition, without contamination from analyte compounds or substances which interfere with analyte compound identification or analysis.
3. For each sampling location (e.g., lake, lake region, stream or river reach), record the location, date, time, collection method, collector, and any other information the collector deems necessary on fish tissue log (Figure 7.15.1).
4. For each fish sampled, record the species, length, weight, and sample identification number on the fish tissue log (Figure 7.15.1). Also, note any anomalies (e.g., lesions, cuts, sores, tumors, fin erosion) observed on the fish.
5. On the left side dorsal area of fish (Figure 7.15.4), clear a small area of scales.
6. Wearing clean latex gloves, insert the 8-millimeter biopsy punch into the fish through the scale free area. The punch is inserted with a slight twisting motion cutting the skin and muscle tissue. Once full depth of punch is achieved a slight bending or tilting of the punch is needed to break off the end of the sample. Remove biopsy punch taking care to ensure sample remains in the punch. Note: The sample should result in a minimum of 0.5 to 0.7 grams of fish tissue for mercury analysis.

7. Apply a generous amount of antibiotic salve to the plug area and gently return the fish to the water.
8. Using a laboratory pipette bulb placed on the end of the biopsy punch, give a quick squeeze, blowing the tissue sample into a sterile 20 milliliter scintillation vial.
9. Dispose of gloves and biopsy punch.
10. Label vial (Figure 7.15.3).
11. Immediately place vial in a plastic bag and put the bag and its contents in a cooler on ice or gel packs.
12. Fill out Sample Identification/Custody/Record form (7.15.2).
13. Place samples in freezer within 48 hours to await analysis.



Figure 7.15.4. Location of plug sample.

9.0 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.
2. Wear latex 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 (7.15.2).
7. If the sample log form indicates a split sample be collected, fill a second sample container and label appropriately (Figure 7.15.3). 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)

10.0 DATA AND RECORDS MANAGEMENT

Fish data will be recorded on the field form 7.15.1 (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.

11.0 QUALITY ASSURANCE AND QUALITY CONTROL

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

12.0 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 7.15.1 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

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

Figure 7.15.2 Fish sample custody form.

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

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

Figure 7.15.3 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.

Document Title:	
Document Revision Number:	
Document Revision Date:	

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]