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QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

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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 fish skin on fillet tissue sample. 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.

Skin on fillet tissue samples is collected for the analysis of contaminants (e.g., pesticides, PCB's, mercury, trace metals) ~~and other contamination~~ to identify health risk if consumed. Due to the sensitive nature of the analysis and potential impact on consumption it is imperative that clean samples are collected using identical protocols each time. A step-by-step guide for the proper collection, preservation, and shipment of these fish tissue samples is provided below. ~~It is developed to ensure the consistency of the collection and the quality of data generated by this program.~~

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.

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 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

Wash and rinse all work surfaces and equipment that will encounter the fish or fillet (e.g., table surface, scaler, filleting knives) between composite samples.

5.0 INTERFERENCES

Transport the samples to a laboratory and keep on ice (not frozen) prior to processing. Each sample must be processed within 48 hours of collection or sample is considered contaminated and is discarded.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel collecting and processing fish skin on fillet 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
- _____ Latex gloves

8.0 FIELD PROCEDURE

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

The following fish species are collected, filleted, and composited for tissue contaminant analysis: walleye, bluegill, sauger, northern pike, bass, crappie, chinook salmon, rainbow trout, catfish, carp, sucker, drum, whitefish, perch, and goldeye.

If available, collect up to five fish of similar predetermined size ranges. Generally, fish are group in sizes ranging from 0-5", 15-20", 20-25", etc. Left-side fillets are collected from each species and size range, as described below in Part 5. One fish is considered acceptable, especially of the larger size ranges.

1. Collect Fish. Several methods of collection are acceptable. The methods ~~most commonly used~~ most used are: 1) electro-fishing; 2) hoop netting; 3) trap netting; 4) gill netting; and 5) hook and line. Any method of collection is acceptable which provides fresh fish in good condition, without contamination from analyte compounds, or substances which interfere with analyte compound identification or analysis.
2. Record on a field data sheet the location, date, time, collection method, collector, and additional information the collector deems necessary (7.14.1).
3. Record fish data on the fish tissue collection data form (Figure 7.14.1). Data is collected from the fish that will be filleted for analysis. -This data should include: 1) species identification; 2) total length; 3) total weight; and 4) notation of anomalous characteristics.
4. Fillet Fish: Wash and rinse all equipment that encounter the fish fillets (e.g., fish scalers, knives, etc.) with soap and water, rinse with clean water, and then acetone. Rinse the equipment between samples ~~which are being submitted for analysis~~.
5. Wash and rinse all work surfaces and equipment which will encounter the fish or fillet (e.g., table surface, scaler, filleting knives) between composite samples.
6. Fish Preparation: All fish, except for Ictalurids (catfish), are scaled prior to filleting. Fish are scaled carefully to not abrade the underlying tissue, thus permitting unnecessary contamination.
7. After scaling has been completed, cut doors ventrally behind the opercular flap from the nape to the top of the rib cage, cutting deep enough to reach the spinal vertebrae. Do not cut into the abdominal cavity. If organs or viscera are cut during the filleting

process, the fillet and equipment are automatically considered contaminated. The fish is discarded, the equipment rinsed, and a new fish is started.

8. Cut posteriorly along the dorsal surface from the opercular cut to the caudal peduncle. Cut deep enough to reach the vertebrae on the anterior portion of the fish. Once past the anus, the knife blade can extend ventrally through the fish. The posterior portion of the fillet is cut following the vertebrae to the caudal peduncle.

9. Returning to the anterior portion of the fillet carefully cut along the top of the rib cage, extracting the bulk of the muscle tissue covering this area. As the muscle tissue thins appreciably, continue cutting downward to the bottom of the fish and then to the exterior. Continue this cut to the caudal peduncle.

10. Place each composite of fish to be analyzed in a resealable plastic bag and write the species, length increment, location, and date on the outside of the bag. Place the sample in a cooler with plenty of ice.

11. Transport the samples to a laboratory and keep on ice (not frozen) prior to processing. Each sample must be processed within 48 hours of collection or sample is considered contaminated and is discarded.

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 (7.14.2).

7. If the sample log form indicates a split sample be collected, fill a second sample container and label appropriately (Figure 7.14.3). 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 7.14.1 (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 skin on fillet 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

Lab ID Number: _____ **Project Code:** _____

Project Description: _____

STORET No.: _____ **Waterbody Name:** _____

Location Description: _____

Date/Time Collected: _____ **Date/Time Processed:** _____

Sampler(s): _____

Collection Method: _____

Species: _____ **Tissue Type:** _____

Comments: _____

Log #	Species Init.	Comp. Size	Sex(m/f/unk.)	Length(cm)	Min	Max	Avg	Mass(g)	Min	Max	Avg
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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):
		Min. Weight (g):	Max. Weight (g):
		Ave. Length (cm):	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 7.13.2 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 7.13.3 Fish flesh label, and fish flesh split label.

APPENDIX B
SOP Acknowledgement and Training Form

