Quality Assurance Project Plan
For Fish Tissue and Water Column Selenium and Mercury

Prepared by:
Aaron Larsen
Watershed Management Program
Division of Water Quality
North Dakota Department of Environmental Quality
Bismarck, ND

Revision 1

April 2021
Effective: 2021 - 2025

This Quality Assurance Project Plan (QAPP) has been prepared to ensure that environmental and related data collected, compiled, and/or generated for this program/project are complete, accurate and of the type, quantity and quality required for their intended use. The work conducted will be in conformance with the Quality Management Plan (QMP) for the Department's Environmental Health Section (NDDoH, March 2016) and with the procedures described in this QAPP. The QMP and this QAPP reflect provisions from the Environmental Protection Agency (EPA) entitled "EPA Requirements for Quality Assurance Project Plans" (March 2001, reissued May 2006).

Approvals:

Aaron Larsen Program Manager Watershed Management Program Division of Water Quality North Dakota Department of Env. Quality Bismarck, ND	Date	Karl Rockeman Director Division of Water Quality North Dakota Department of Env. Quality Bismarck, ND	Date
Dennis Fewless Quality Assurance Coordinator North Dakota Department of Env. Quality Bismarck, ND	Date		

REVISION PAGE

Date	Revision #	Summary of Changes	Sections	Other Comments
04/5/2021	0	n/a	n/a	Document creation

Revision Page ii

Table of Contents iii

List of Tables iv

List of Figures v

Distribution List vi

A Project Management

A1 Title and Approval Sheet 1

A2 Table of Contents 1

A3 Distribution List 1

A4 Project/Task Organization 1

A5 Problem Definition/Background 3

A6 Project Monitoring Goals/Objectives/Tasks Description 7

A7 Data Quality Objectives and Criteria for Measurement Data 9
A7.1 Data Quality Objectives 9
A7.2 Measurement Performance Criteria 10

A8 Special Training/Certification 15

A9 Documents and Records 15

B Data generation and acquisition

B1 Sampling Process Design (Experimental Design) 16
B1.1 Monitoring Goals 16
B1.2 Sampling Locations 17
B1.3 Sampling Frequency 18

B2 Sampling Methods 18

B3 Sample Handling and Custody Requirements 19

B4 Analytical Method Requirements 20

B5 Quality Control 20

B6 Instrument/Equipment Testing, Inspection and Maintenance 21

B7 Instrument/Equipment Calibration and Frequency 21

B8 Inspection/Acceptance of Supplies and Equipment 21

B9 Data Management 22

C Assessment and oversight

C1 Assessment and Response Actions 22

C2 Reports to Management 24

D Data validation and usability

D1 Data Review, Validation and Verification Requirements 24

D2 Verification and Validation Methods 24

D3 Reconciliation with Data Quality Objectives and User Requirements 25

Literature Cited 25

Appendices

A Standard operating procedure (SOP) for Measuring Secchi Disk Transparency

B SOP for Taking Field Measurements in Streams, River, Lakes and Wetlands Using Handheld Meters

C SOP for Collecting and Preserving Lake Water Quality Samples

D SOP for Collection and Processing of Fish Tissue Plug Samples for Mercury Analysis

E SOP for Collecting and Preserving Whole Fish Tissue Samples

F SOP for Collecting and Preserving Stream and River Grab Samples

List of Tables

Table 1 Lakes and Reservoirs Selected for the 2021 Fish Tissue and Water Column Selenium and Mercury Project 4

Table 2 Sampling Date Ranges for the 2021 Fish Tissue and Water Column Selenium and Mercury Project 7

Table 3 2021 Fish Tissue and Water Column Selenium and Mercury Project Data Needs and Intended Use 10

Table 4 Measurement Performance Criteria For Laboratory Analytes 12

Table 5 Summary of Precision, Accuracy and Completeness Requirements 14

Table 6 Samples to be Collected With Analyte Number, Key Parameters, Bottle, Preservation Method and Holding Time 15

Table 7 Total Number of Samples to be Collected by Type During the 2021 Fish Tissue and Water Column Selenium and Mercury project 17

Table 8 Sampling Methods 18

Table 9 List of Potential Fish Species for River and Stream Sites 19

List of Figures

Fig. 1 Organizational Diagram for the 2021 Fish Tissue and Water Column Selenium Project 2

Fig. 2 Location of Potential River and Stream Sites Selected for the 2021 Fish Tissue and Water Column Selenium Project 5

Fig. 3 Location of Potential Lakes and Reservoirs Selected for the 2021 Fish Tissue and Water Column Selenium Project 6

Distribution List

Joshua Baker
Monitoring Coordinator
US EPA Region 8
1595 Wynkoop Street
Denver, CO 80202

Mary Goldade
Deputy Regional QA Manager
Quality Assurance Branch
US EPA Region 8
1595 Wynkoop Street
Denver, CO 80202

Anthony Deloach
EPA Region 8 PPA/G Coordinator
US EPA Region 8
1595 Wynkoop Street
Denver, CO 80202

Aaron Larsen
Program Manager/Designated Project Manager
Watershed Management Program
North Dakota Dept. of Environmental Quality
918 E. Divide Ave, 4th Floor
Bismarck, ND 58501

Karl Rockeman
Director
Division of Water Quality
North Dakota Dept. of Environmental Quality
918 E. Divide Ave, 4th Floor
Bismarck, ND 58501

Joshua Wert
Environmental Scientist/Field Investigator
Division of Water Quality
North Dakota Dept. of Environmental Quality
918 E. Divide Ave, 4th Floor
Bismarck, ND 58501

Peter Wax
Special Projects Coordinator
Division of Water Quality
North Dakota Dept. of Environmental Quality
918 E Divide Ave, 4th Floor
Bismarck, ND 58501

Dennis Fewless
Quality Assurance Coordinator
NDDEQ Chief's Office
918 E. Divide Ave
Bismarck, ND 58501

A. Project Management

A1. Title and Approval Sheet

See Page i.

A2. Table of Contents

See Page iii.

A3. Distribution List

See Page vi.

A4. Project/Task Organization

This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) activities/procedures that will be used while collecting samples for the 2021 Fish Tissue and Water Column Selenium and Mercury. The purpose of this document is to present the standardized methods and procedures that will be used to collect chemical and biological samples and measurements from rivers and streams as well as lakes and reservoirs within North Dakota to ensure quality data. The QAPP also describes the duration, scope and frequency of monitoring. Data will be collected by the North Dakota Department of Environmental Quality staff.

The organization for the North Dakota Department of Environmental Quality (NDDEQ) is detailed in the Quality Management Plan (QMP) for the Environmental Health Section (NDDoH, 2016)[1]. Within the NDDEQ there are five divisions, including the Divisions of Air Quality, Municipal Facilities, Waste Management, Water Quality and Laboratory Services. Dennis Fewless is the Quality Assurance Coordinator (QAC) for the NDDEQ. The QAC is located in the NDDEQ Chief's Office and reports directly to the Chief of the NDDEQ. The NDDEQ Chief's Office, through the QAC, is responsible for oversight of the NDDEQ's quality system for QA and QC as delineated in its QMP, including approving project QAPPs. It is the policy of the NDDEQ that the primary responsibility for QA resides among program staff and Designated Project Managers (DPMs) in each division; therefore, each program is responsible for the preparation, implementation and assessment of its QAPP(s).

Within the NDDEQ, the Division of Water Quality is organized in four programs: the North Dakota Permit Discharge Elimination System (NDPDES) Program, the Groundwater Program, Watershed Management Program (WMP) and special projects. The organizational structure for the Fish Tissue and Water Column Selenium and Mercury project is outlined in Figure 1.

[1] This QAPP was prepared according to the EHS's QMP, which has been approved by EPA.

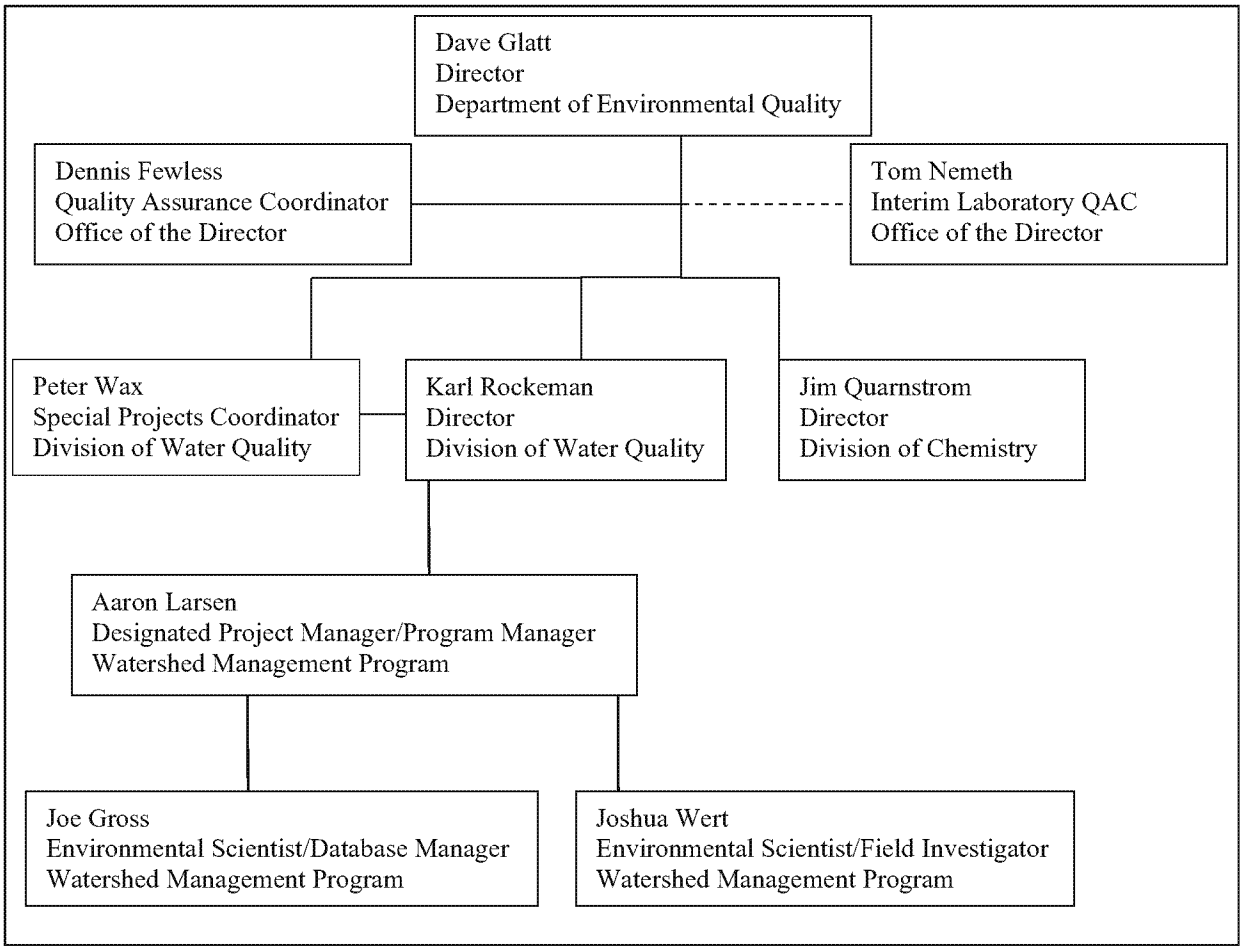

Figure 1. Organizational Diagram for the 2021 Fish Tissue and Water Column Selenium and Mercury Project.

Aaron Larsen is Program Manager (PM) for the WMP. As Program Manager in the WMP he has the following responsibilities:

- Review and approve the QAPP;
- Provide oversight for study design, site selection and adherence to design objectives;
- Review and approve the final project work plan and other materials to support the project (e.g., Standard Operating Procedures - SOPs);
- Select appropriate project subcontractors, as needed; and
- Coordinate with contractors, reviewers and EPA to ensure technical quality and contract adherence.

Aaron Larsen is also the Designated Project Manager (DPM) for the 2021 Fish Tissue and Water Column Selenium and Mercury Project (Figure 1). As such, he is responsible for overall project coordination and supervision of data collection, as well as overseeing corrective actions (as needed). Joshua Wert is an Environmental Scientist with the WMP and is the primary Field Investigator (FI) for the project (Figure 1). As the FI, Mr. Wert is responsible for writing and maintaining the QAPP (as needed), river/stream and lake/reservoir field sampling, chain of custody, data analysis, records management and reporting for the 2021 Fish Tissue and Water Column Selenium and Mercury project. Joe Gross will be responsible for data management and storage. Further, Mr. Wert is responsible for performing and documenting data quality in accordance with Data Quality Objectives (DQOs) outlined in Section A4.

An organizational diagram for the EPA Region 8 laboratory is provided in U. S. EPA Region 8 Environmental Laboratory Quality Assurance Manual. US EPA Region 8 Laboratory, Lakewood, CO. (*In revision*).

A completed QAPP must be signed by the Director of the Division of Water Quality, Karl Rockeman, as well as the DPM, Aaron Larsen. Final approval of a completed or revised QAPP is done by the QAC, Dennis Fewless.

A5. Problem Definition/Background

It is the responsibility of the NDDEQ to monitor and assess the state's water resources (with the exclusion of water's on Native American lands) for the benefit of the people of North Dakota and the Nation. Pollutant loads are ever-increasing across waters of the State and selenium can be released into waters via natural runoff or various human activities such as mining, irrigation, etc. Even though selenium is an essential element, it can become toxic to animals/wildlife in high amounts or concentrations. Biological monitoring efforts in 2021 will focus on rivers and streams in the James and Souris river basins (eco region 46). This project will assess selenium concentrations in the water column to determine if there is a direct link to selenium concentrations in the fish tissue associated with aquatic life inhabiting North Dakota waters. Additionally, data from this project will determine what selenium water quality criterion is necessary to protect aquatic life in North Dakota.

Table 1. Potential Lakes and Reservoirs Selected for the 2021 Project Fish Tissue and Water Column Selenium and Mercury Project.

Lake Name	County	Basin	STORET Number	Latitude	Longitude
Barnes Lake[7,8]	Stutsman	James	380995	47.23421	-99.27754
Baukol-Noonan Dam[7,8]	Divide	Souris	381320	48.86915	-102.95269
Buffalo Lodge Lake[2,7]	McHenry	Souris	383005	48.33104	-100.76269
Clark Lake[7,8]	Stutsman	James	381155	47.17625	-99.41524
George Lake[7,8]	McHenry	Souris	383000	48.12347	-100.42086
Hehn-Schaffer Lake[7,8]	Stutsman	James	385568	46.68415	-99.13381
Hurdsfield-Tuffy Lake[2,3]	Wells	James	385598	47.45591	-99.8663
Jamestown Reservoir[2,3,4,6,7,8]	Stutsman	James	381165	46.93256	-98.70822
			385463	46.98765	-98.7112
			385005	47.01817	-98.73688
			385007	47.07202	-98.75538
Kalmbach Lake[7,8]	Lamoure	James	380780	46.50478	-98.9902
Kulm-Edgeley Dam[7,8]	Lamoure	James	380790	46.32949	-98.81638
Lake Darling[1,3,6,7]	Renville	Souris	384140	48.45917	-101.58568
			384141	48.591	-101.61041
			384142	48.6669	-101.6971
Lake Juanita[4,7,8]	Foster	James	381070	47.55922	-98.72667
Lake Lamoure[2,3,4,7]	Lamoure	James	380795	46.3002	-98.2708
Lake Metigoshe[2,3,7,8]	Bottineau	Souris	380610	48.99634	-100.35851
			380611	48.96425	-100.34978
			380612	48.97765	-100.35357
Moores Lake[7,8]	Dickey	James	385567	46.03484	-98.87907
Northgate Dam[2,4,5,7]	Burke	Souris	380845	48.92429	-102.26945
Pheasant Lake[3,4,5,7,8]	Dickey	James	381125	46.00527	-98.67451
Round Lake[7,8]	McHenry	Souris	385512	48.03294	-100.27643
Short Creek Dam[4,5,7]	Burke	Souris	380905	46.34096	-101.84853
Twin Lakes[2,3,8]	Lamoure	James	386035	46.40253	-98.264
Velva Sportsman's Dam[7,8]	Ward	Souris	381350	47.93575	-100.9715

[1]NDGF Tier 1 Fishery
[2]NDGF Tier 2 Fishery
[3]HABs Advisory in past
[4]Past or present 319 project
[5]Approved TMDL
[6]On 303(d) list
[7]In Water Quality Standards
[8]Greater than or equal to ten years since last sample or no historical sample

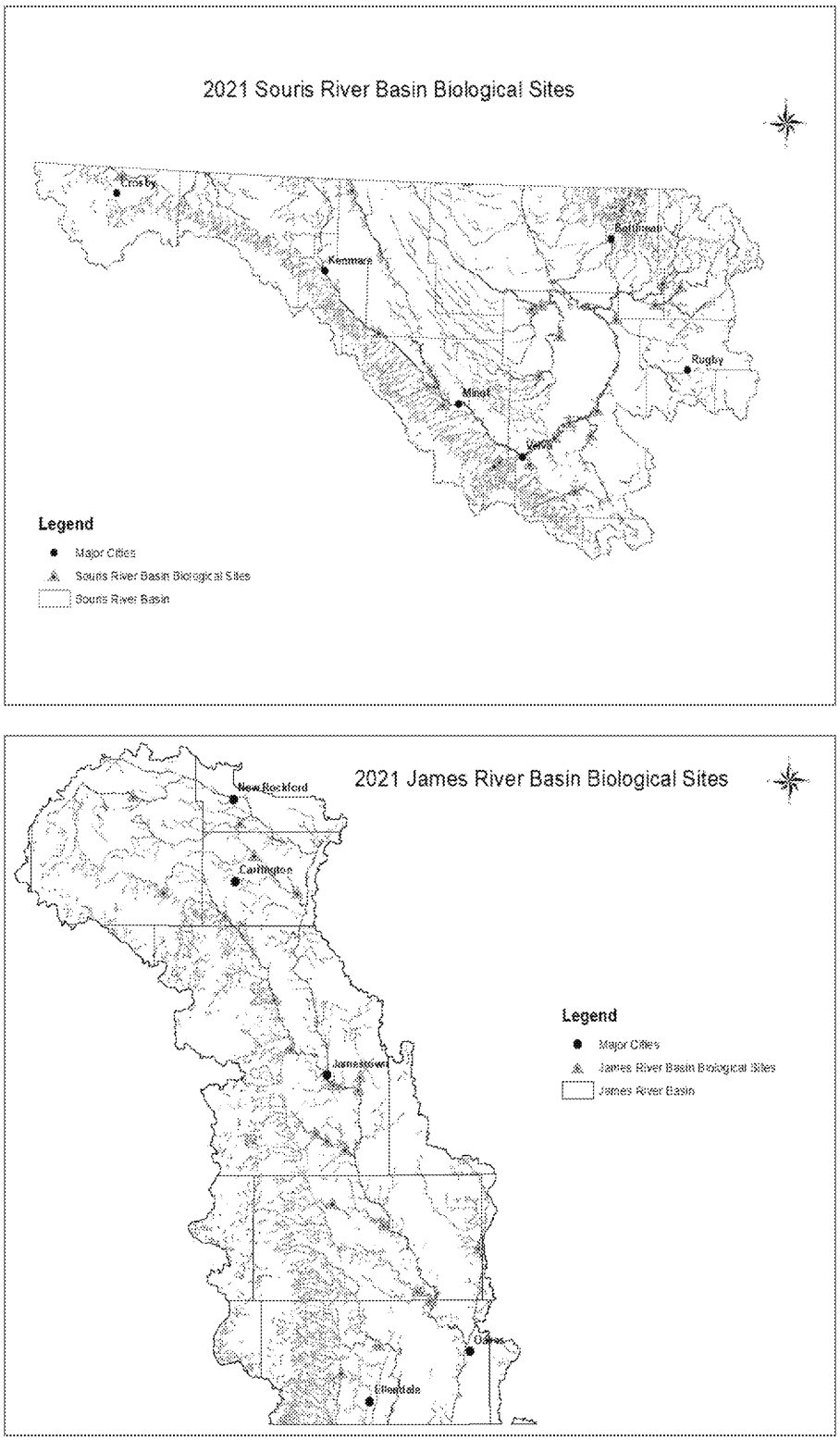

Figure 2. Location of Potential River and Stream Sites Selected for the 2021 Fish Tissue and Water Column Selenium and Mercury Project in the Souris (top) and James (bottom) River Basins.

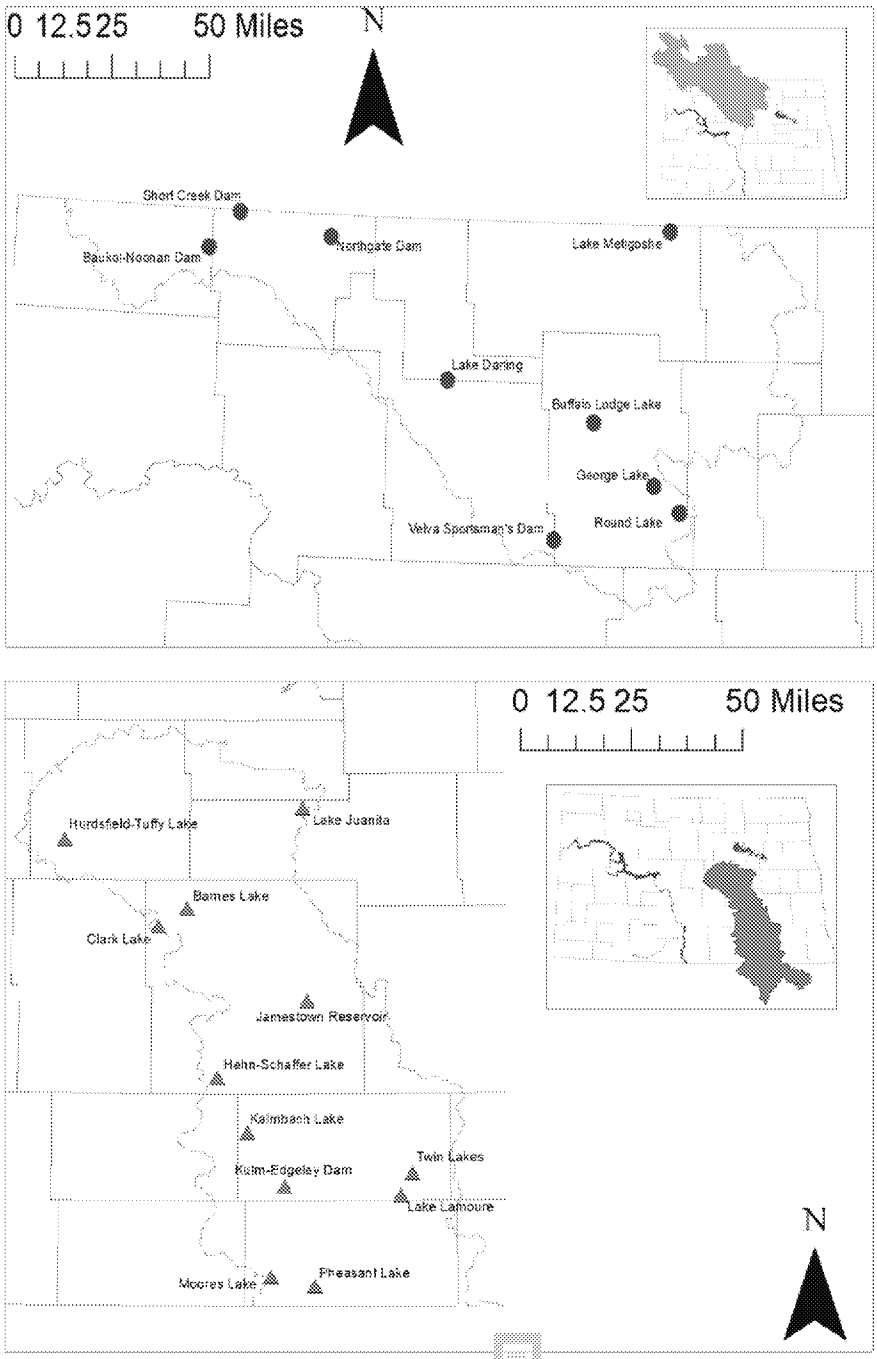

Figure 3. Location of Potential Lakes and Reservoirs Selected for the 2021 Fish Tissue and Water Column Selenium and Mercury Project in the Souris (top) and James (bottom) River Basins.

Table 2. Sampling Date Ranges for the 2021 Fish Tissue and Water Column Selenium and Mercury Project.

Sampling Period	Date Ranges for Sampling Periods
Rivers and Streams	June 1 – August 15
Lakes and Reservoirs	August 16 – September 30

A6. Project Monitoring Goals/Objectives/Tasks Description

The goal of the 2021 Fish Tissue and Water Column Selenium and Mercury project is to assess the extent of selenium as a concern to North Dakota lakes, reservoirs (Table 1 and Figure 3), rivers, and streams (Table # and Figure 2). In addition to assessing the extent of selenium in the water column, this project will also evaluate the extent of bioaccumulation of selenium in fish tissue. Biological monitoring associated with the Reference River and Stream Site Monitoring and Lake Water Quality Assessment (LWQA) sampling efforts will focus on the James and Souris River basins in 2021. Field data collection will include in-situ measurements (temp, DO, pH and specific conductance) at each river and stream location. In lakes and reservoirs, an entire profile will be recorded throughout the water column. Further information associated with the NDDEQ's biological monitoring and LWQA projects can be found in the Reference River and Stream Site Monitoring and LWQA project specific QAPPs (NDDEQ 2021).

Assessment information generated from this project could be used by both governmental agencies and citizens of North Dakota. The agencies that are most likely to utilize the data are the NDDEQ and the North Dakota Game and Fish Department (NDGF). Members of the public most likely to utilize the data include local residents, anglers and lake residents.

Identified uses of the data include the prioritization of lakes, reservoirs and their watersheds for lake maintenance and improvement projects (i.e., Save Our Lakes (SOL), Total Maximum Daily Loads-TMDL, Section 319 Non-point Source Management Program) and to assess the condition of the state's waterbodies for Section 305(b) water quality assessment purposes.

The NDDEQ will use data collected during the 2021 Fish Tissue and Water Column Selenium and Mercury project to update the 303(d) list of impaired waters, when necessary. Data will be evaluated to determine if state water quality standards for selenium are adequate in order to protect aquatic life (available at Water Quality Standards) and determine if bioaccumulation is a concern for North Dakota citizens who routinely consume fish tissue.

The following objectives and tasks are intended to achieve the monitoring goals of the project. Specific milestones and products are provided with each task.

Objective 1: Collect chemical and biological data from the 20 river and stream sites associated with North Dakota's Reference River and Stream Site Monitoring Program. Exact sampling locations are currently being evaluated and will be finalized prior to data collection.

Task 1: Once in June/July, collect from each sampling site, a dissolved oxygen, temperature, specific conductance, and pH reading (Appendix B).

Product: Data sets of temperature, dissolved oxygen, pH and specific conductance profiles collected from each river and stream sampling site.
Milestone: October 31, 2021

Task 2: Once in June/July, collect fish tissue and water chemistry (metals) data at 20 river and stream sites (Figure 2). Fish tissue selenium composite samples for small fish less than 200 mm (i.e., fathead minnows, darter spp., etc) will ideally include 5 individuals per species but 3 will suffice as a minimum. If more than 5 species present at a site, collect as many 3-5 fish composites as possible. For fish greater than 200 mm, 2 fish per species will be composited if they meet the 75% rule.

Product: Fish tissue and water column selenium data from each river and stream sampling site. Fish tissue selenium samples will be sent to EPA R8 laboratory for analysis (EPA Region 8 QAPP).
Milestone: April 1, 2022

Task 3: Once in June/July, collect fish tissue plugs for mercury analysis from commonly consumed sportfish encountered in rivers and streams (i.e., opportunistic). Based on the species listed in Table 9, fish tissue plugs will be collected from walleye, yellow perch, northern pike, bluegill, and crappie spp.

Product: Opportunistic fish tissue plugs for mercury analysis from consumable North Dakota fish species.
Milestone: April 1, 2022

Task 4: Once in late August/September, collect a dissolved oxygen, temperature, specific conductance, and pH profile from a subset of lakes/reservoirs listed in Figure 2.

Product: In-situ data collected from a subset of lakes/reservoirs in Table 1.
Milestone: October 31, 2021

Task 5: Once in late August/September, utilizing a 6-foot integrated water column sampler, collect water chemistry samples from a subset of lakes/reservoirs in Table 1.

Product: Data sets of water chemistry parameters listed in Table 4.
Milestone: October 31, 2021

Task 6: Once in late August/September, collect fish tissue selenium data from a subset of lakes/reservoirs.

Product: Data sets of fish tissue selenium data
Milestone: March 2022

Task 7: Compile and analyze all QA/QC data collected throughout the project period (i.e., duplicate and blanks). All data will be compiled into a brief report to be attached to the final report as an appendix.
Product: Final QA/QC report
Milestone: March 2022

Objective 2: Ensure the data are readily available to natural resource professionals (e.g, fisheries managers, hydrologists, engineers, water quality specialists) and the public.

Task 8: Enter and store all data collected during the Fish Tissue and Water Column Selenium and Mercury project in the WMP's Access 2000 Sample Identification Database (SID) and EPA's WQX database.
Product: Project data entered in SID and WQX.
Milestone: March 2022

Task 9: Compile and analyze all QA/QC data collected throughout the project period (i.e., duplicate and blank samples). All data will be compiled into a brief report to be attached to the final report as an appendix.
Product: Final QA/QC report.
Milestone: March 2022

A7. Data Quality Objectives and Criteria for Measurement Data

A7.1 Data Quality Objectives

It is the policy of the EPA and the NDDEQ that data quality objectives (DQOs) be developed for all environmental data collection activities. Data of known quality are essential to the success of any monitoring or sampling project. DQOs are qualitative and quantitative statements that clarify the intended use of the data, define the type of data needed to support the decision, identify the conditions under which the data should be collected, and specify tolerable limits on the probability of making a decision error due to uncertainty in the data. DQOs are developed by data users to specify the data quality needed to support specific decisions. Sources of error or uncertainty include the following:

- Sampling error: The difference between sample values and *in situ* true values from unknown biases due to collection methods and sampling design;
- Measurement error: The difference between sample values and *in situ* true values associated with the measurement process;
- Natural variation: Natural spatial heterogeneity and temporal variability in population abundance and distribution; and

- Error sources or biases associated with compositing, sample handling, storage and preservation.

This assessment uses a "fixed station design" as outlined in the state's monitoring strategy (NDDoH, 2014) (available at Monitoring Strategy). This design can be used to detect "trends" in water quality, and while true trend analysis can not be achieved with the data as collected (given the time between sampling years in most cases), comparability over time using the data collected and historical data can still be achieved qualitatively. Further, this sampling method allows the WMP to rapidly assess the trophic state of the state's lakes and reservoirs. Methods and procedures described in this document are intended to reduce the magnitude of the sources of uncertainty (and their frequency of occurrence) by applying the following approaches:

- Use of standardized sample collection, handling and analysis procedures; and

- Use of trained scientists and technicians to perform the sample collection and handling activities.

A7.2 Measurement Performance Criteria

In order to meet the DQO for the project, the types of data needed for this project and their intended use are described in Table 3. For each of these data, a discussion of the measurement performance criteria or data quality indicators is provided. Data quality indicators include the following:

- Precision;
- Accuracy;
- Representativeness;
- Completeness; and
- Comparability.

Table 3 2021 Fish Tissue and Water Column Selenium and Mercury Project Data Needs and Intended Use.

Data Needed	Intended Use
Secchi disk transparency measurements	To provide an estimate of trophic status through Carlson's Trophic Status Index using Secchi Disk Transparency measurements.
Temperature and dissolved oxygen (DO) profile measurements	To identify oxygen abundance or deficiency, and the thermal/chemical stratification regime of the lake or reservoir (i.e., depth of epilimnion, metalimnion and hypolimnion) per visit.

Data Needed	Intended Use
Chemical and biological water quality monitoring data	To characterize the lake or reservoirs current water quality condition and trophic status, and assess possible spatial differences in water quality and trophic status among lakes, reservoirs and ecological regions.

Measurement Performance Criteria for Laboratory Analytes sampled for this project are provided in Table 4. Measurement performance criteria are provided for all chemical analytes sampled and analyzed as part of this project. Measurement performance criteria are provided to ensure that the "achievable laboratory limits" (i.e., method detection and quantification limits) provided by the laboratory performing the analysis are consistent with the projects quantification limits and/or action limits. Project quantification limits cannot always be met due to numerous factors (the most prevalent in North Dakota being interference due to sediment). If the quantification limit cannot be achieved by the laboratory due to some kind of interference, it will be at the discretion of the DPM as to whether or not additional sampling or a change in sampling method needs to occur; the latter being satisfied by dissolving metals for a lake instead of using an unfiltered sample to eliminate any bias or interference from sediment.

Precision is a measure of mutual agreement among individual measurements or enumerated values of the same property of a sample, usually under demonstrated similar conditions. Precision is best measured in terms of the standard deviation. For purposes of this project, precision of biological samples and chemical analysis will be calculated from replicate samples and expressed as relative percent difference (RPD), if it is calculated from duplicate samples, or as relative standard deviation (RSD), if it is to be calculated from three or more samples. Table 5 provides a summary of the precision requirements for data collected for this project. To calculate RPD, the equation used is

$$\mathbf{RPD = (C_D - C_S) / C_S}$$

where C_D stands for Duplicate Concentration and C_S is Sample Concentration. The FI and DPM will compare RPD to the criteria in Table 5. Samples that are found to be outside of the RPD criteria will be evaluated on a case-by-case basis. For example, some analytes are difficult to achieve high levels of precision (e.g., chlorophyll-a) and their deviation from the criteria will be considered. Some samples may be considered a field error based on notes made in the field regarding sample collection and preparation. Finally, the lab may be consulted if there is a possibility of transcription error or any other laboratory error. Data will be flagged and commented based on findings from precision samples.

Table 4. Measurement Performance Criteria for Laboratory Analytes.

Analyte	Matrix	Action Limit	Project Quantification Limit (QL)	Analytical Method	Achievable Laboratory Limit	
					MDLs	QLs
Aluminum	Water	87 µg/L[11]	50 µg/L	200.7[5]	7.700 µg/L	50 µg/L
Antimony	Water	5.6 µg/L[5,9]	1 µg/L	200.8[5]	0.004 µg/L	1.00 µg/L
Arsenic	Water	10 µg/L[5,9]	1 µg/L	200.8[5]	0.040 µg/L	1.00 µg/L
Barium	Water	1,000 µg/L[6]	1 µg/L	200.8[5]	0.008 µg/L	1.00 µg/L
Beryllium	Water	4 µg/L[5,9]	1 µg/L	200.8[5]	0.030 µg/L	1.00 µg/L
Bicarbonate	Water	NA	4 mg/L	2320 B[3]	NL[2]	4 mg/L
Boron	Water	750 µg/L[7]	50 µg/L	200.7[5]	6.700 µg/L	50 µg/L
Cadmium	Water	0.72 µg/L[8,11]	1 µg/L	200.8[5]	0.020 µg/L	1.00 µg/L
Calcium	Water	NA	2 mg/L	200.7[5]	0.009 mg/L	2.00 mg/L
Carbonate	Water	NA	1 mg/L	2320 B[3]	NL[2]	1 mg/L
Chloride	Water	100 mg/L[5,7]	1 mg/L	300.0[5]	0.370 mg/L	1 mg/L
Chromium	Water	NA	1 µg/L	200.8[5]	0.030 µg/L	1.00 µg/L
Conductivity	Water	NA	NA	2510 B[3]	NA	NA
Copper	Water	9.3 µg/L[8,11]	1 µg/L	200.8[5]	0.40 µg/L	1.00 µg/L
Fluoride	Water	4 mg/L[5,9]	0.050 mg/L	300.0[5]	0.021 mg/L	0.05 mg/L
Hardness	Water	NA	NL[2]	2340 B[3]	NL[2]	NL[2]
Hydroxide	Water	NA	1 mg/L	2320 B[3]	NL[2]	1 mg/L
Iron	Water	NA	0.05 mg/L	200.7[5]	0.014 mg/L	0.05 mg/L
Lead	Water	3.2 µg/L[8,11]	1 µg/L	200.8[5]	0.33 µg/L	1.00 µg/L
Magnesium	Water	NA	1 mg/L	200.7[5]	0.251 mg/L	1.00 mg/L
Manganese	Water	NA	0.01 mg/L	200.7[5]	0.005 mg/L	0.01 mg/L
Molybdenum	Water	NA	1 µg/L	200.8[5]	0.32 µg/L	1.00 µg/L
Nickel	Water	52 µg/L[8,11]	1 µg/L	200.8[5]	1.63 µg/L	1.00 µg/L
pH	Water	7.0 – 9.0[5]	NL[2]	4500 H+ B[3]	NL[2]	NL[2]
Potassium	Water	NA	1 mg/L	200.7[5]	0.282 mg/L	1.00 mg/L
Selenium	Water	5 µg/L[8,11]	1 µg/L	200.8[5]	0.58 µg/L	1.00 µg/L
Silica	Water	NA	2 mg/L	200.7[5]	0.13 mg/L	2 mg/L
Silver	Water	3.8 µg/L[8,10]	1 µg/L	200.8[5]	0.27 µg/L	1.00 µg/L
Sodium	Water	NA	3 mg/L	200.7[5]	2.61 mg/L	3.00 mg/L
Sulfate	Water	250 mg/L[5,7]	1 mg/L	300.0[5]	0.21 mg/L	1.00 mg/L
Thallium	Water	0.24 µg/L[5,9]	1 µg/L	200.8[5]	0.43 µg/L	1.00 µg/L
Total Dissolved Solids	Water	NA	NL2	1030 F3	NL2	NL2
Zinc	Water	120 µg/L8,10,11	1 µg/L	200.85	0.52 µg/L	1.00 µg/L

[1] Method Detection Limit
[2] Not Listed in the Method.
[3] From Standard Methods, 19th Edition
[4] From Standard Methods, 20th Edition
[5] US EPA Clean Water Act Method Approved for Use at 40 CFR 136
[6] Based on 1-day arithmetic average

[7]Based on 30-day arithmetic average
[8]This is a hardness-dependent criteria (value given based on hardness of 100 mg/L)
[9]Based on human health criteria
[10]Based on acute aquatic health criteria
[11]Based on chronic aquatic health criteria
[12]Based on a pH of 9, temperature of 30, and where salmonids are present

Accuracy is the degree of agreement between an observed or measured value and the true or expected value of the measured quality. Many kinds of error, including unintentional bias affect the inherent accuracy of data. A **bias** can be negative or positive or neither. For example, a positive bias in total nitrogen samples would be using evidence to suggest that total nitrogen is being added to samples independent of the sample being collected. While the investigator almost never knows true population values, accuracy of chemical laboratory samples may be obtained from the analysis of spiked matrix samples. Performance evaluation (PE) samples may also be employed to estimate the accuracy of chemical analysis. This is especially true when working with natural biological communities. Therefore, the best an investigator can do is to avoid bias by assuring consistency of sampling and sample processing and striving for repeatability of measurements. Table 5 provides a summary of the accuracy requirements for data collected for this project. Also, NDDEQ uses blank samples to ensure that the sampling and processing of a sample doesn't bias sample results. There is no calculation that is done for this, but instead staff check laboratory data and note when there are analytes that have documented concentrations instead of a "non-detect", also known as a "hit". These hits will be documented by the FI and DPM, and remediation of this result will depend on the analyte and the magnitude of the error.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter, variation at a sampling point, process condition, or an environmental condition. The representativeness of the project relies in part on the selection of sample sites and the collection of a significant number of samples. This assessment uses a "fixed station design" as outlined in the state's monitoring strategy (NDDoH, 2014) (available at Monitoring Strategy).

Completeness is defined as the percentage of measurements made that are judged to be valid according to specific criteria and entered into the data management system. To optimize completeness, every effort is made to avoid sample and/or data loss. Accidents during sample transport or lab activities that cause the loss of the original samples will result in irreparable loss of data, which will reduce the ability to perform analysis, integrate results, and prepare reports. In order to maximize completeness, all samples will be stored and transported in unbreakable (plastic) containers when possible.

Percent completeness (%C) for measurement parameters and samples is defined as:

$$\%C = v/T \times 100$$

Where v = the number of measurements or samples judged valid; and
T = the total number of measurements of samples collected.

Samples will be collected at 100% of the sites unless access is denied or is physically impracticable. In the case of unanticipated unsafe conditions (i.e., bad weather) sampling will be postponed until a later date. Table 5 provides a summary of the completeness requirements for data collected for this project. The end-product for the Fish Tissue and Water Column Selenium and Mercury project is informing of the state's 303(d) list of impaired waters along with assessing the extent of selenium in both the water column and fish tissue. If completeness requirements are not met for parameters and/or lakes, it will be the decision of DPM if additional sampling is needed to inform listing or de-listing of waterbodies.

Natural variability is to be expected within a waterbody, even within the same day or same sample. Variability in sample results can be caused by natural conditions (wind, sun), not properly storing sample prior to processing (time, light), or human error (not wearing gloves), among others. Following documented SOPs, however, will help to minimize sampler error and increase precision and accuracy of samples being collected. Collecting QA samples in succession with the original sample will help maximize precision and accuracy.

Table 5. Summary of Precision, Accuracy and Completeness Requirements.

Measurement Parameter	Precision	Accuracy	Percent Completeness
Secchi disk transparency (lakes only)	0.05 meter	0.1 meter	95 %
Water chemistry (e.g., general chemistry, trace metals)	20 %	NA	95 %
Field profile measurements			
Dissolved oxygen (DO)	0.1 mg/L	0.3 mg/L	95 %
Temperature	0.1 °C	0.2 °C	95 %
pH	0.1 units	0.2 units	95 %
Specific conductance	1 µmohs	10 µmohs	95 %

Comparability is a measure of the confidence with which one data set can be compared to another. Comparability is dependent on the proper design of the sampling program and on strict adherence to accepted sampling techniques, standard operating procedures and quality assurance guidelines. For this project, comparability of data will be accomplished by standardizing the sampling season, the geographic extent of the project, the field sampling methods and the field training as follows:

- Standard sampling and analytical methods, as well as standard units of reporting for all parameters sampled, will be used (Appendices A-F).
- All field personnel involved with sampling will have adequate training and experience. Any relevant training activities occur prior to water quality sampling in May and will be documented by the WMP.

Table 6. Samples to be Collected with Analyte Number, Key Parameters, Bottle, Preservation Method and Holding Time.

Analyte name	Analyte number	Key parameter(s)	Bottle	Preservation	Holding time
Trace metals - dissolved	144	Selenium	250 ml Nalgene	Nitric Acid and Cool to 4°C	7 days

A8. Special Training/Certification

The FI will be responsible for all field data collection. The FI and those assisting in field sampling are required to have the necessary knowledge and experience to perform all field activities. Training in the proper methods for sample collection, preservation and the transfer of water chemistry will be provided by the FI.

All samplers will be trained by the FI using SOPs applicable to this project (all noted in Table 9). Each SOP now has a signature page for acknowledgement that each sampler has read and understands the SOP and has been trained in the field on each sampling procedure. These sampler trainings can either be performed one-on-one or in a group. Each sampler will sign this page prior to participating in the Fish Tissue and Water Column Selenium and Mercury project. A hard copy of the signature page will be kept by the FI and kept electronically on the Y drive.

A9. Documents and Records

Following the receipt of all data from the laboratory, two-page fact sheets will be produced for each lake. These fact sheets will have information on land cover, temperature and dissolved oxygen profiles, trophic state indices, nutrients and water quality. Fact sheets will be published on the program's website upon completion (available at Publications). Also, the program will develop annual lake water quality reports where all data collected during the project will be summarized for each lake and published at the site above. Finally, attached to the end of this report as an appendix will be a summary of QA data for the project.

Thorough documentation of all field sampling and handling activities is necessary for proper processing in the laboratory, data reduction and, ultimately, for the interpretation of study results. Field sample collection and handling will be documented in writing using the following forms and labels:

- A set of Sample Identification/Custody Record forms will accompany each water chemistry sample submitted to the Division of Laboratory Services-Chemistry laboratory for analysis (Appendix C):

- A sample identification label will be affixed to each sample collected and submitted to the Division of Laboratory Services-Chemistry laboratory (Appendix C); and

- All field measurements (i.e., weather conditions, Secchi disk transparency, temperature, DO, pH and specific conductance measurements) will be recorded on the Lake and Reservoir Field Data Form (Appendix B). River and stream field measurements will be recorded in the River and Stream Field Log (Appendix E).

All these forms will be stored in files at the Gold Seal Building by the FI. Other documents to be produced and stored (but not necessarily circulated) will be calibration logs, sample logs and copies of custody forms sent to EPA Region 8 laboratory.

Each sample collected will be uniquely identified on the sample label and field custody forms by specifying the sample: ID, location, date, time and the name of the sampler.

Project information records are kept at the Gold Seal Building in binders for 10 years. At the end of this period, hard copy records are filed in bank boxes and stored at a storage shed near the laboratory ("cold storage") for an indefinite period. These records will not be discarded. Electronic files stored on network drives are backed-up daily by the North Dakota Information Technology Department (ITD).

Prior to sampling in 2021, a copy of the approved QAPP will be distributed (by email) to everyone on the distribution list on page vi.

B. Data Generation and Acquisition

B1. Sampling Process Design (Experimental Design)

B1.1 Monitoring Goals

The goal of the 2021 Fish Tissue and Water Column Selenium and Mercury project is to identify, through water quality sampling and fish tissue collection, the extent of selenium concentrations in North Dakota surface waters along with its impacts on aquatic life (i.e., bioaccumulation, etc). The waterbodies selected for 2021 are based on existing monitoring projects such as the Lake Water Quality Assessment Project (LWQA) and the Reference River and Stream Site Monitoring Project. The 2021 Fish Tissue and Water Column Selenium and Mercury project focuses sampling efforts in the James and Souris River basins.

Assessment information generated from this project is designed to be used by both governmental agencies and citizens of North Dakota. The agencies that are most likely to utilize the data are the NDDEQ and NDGF. Member of the public most likely to utilize the data include local residents, anglers and lake residents.

Identified uses of the data is the prioritization of lakes, reservoirs and their watersheds for lake maintenance and improvement projects (i.e., Save Our Lakes-SOL, Total Maximum Daily Loads-TMDL, Section 319 Non-point Source Management Program) and to continue and improve the monitoring and assessment of the state's and nation's waters (i.e., Section 305(b)).

Distribution of the monitoring results is not limited and other groups likely to utilize some or all of the data include political sub-divisions, concerned lake users, lake associations, students, county soil conservation districts and water resource boards.

Most information recorded by the FI during sampling should be considered "critical" information. While weather conditions (e.g., temperature, cloud cover, wind) may not be used in analysis and is more qualitative than quantitative, it may help in understanding the data. Recording things like if there was a lot of boat activity on the lake, if the ramp was inaccessible, or if the road was impassible are examples of informational data. These are not critical to the understanding of the data, but it may be useful to know for future sampling efforts.

In the field, sampling staff can encounter situations where the sampling sites become inaccessible. While an inaccessible sampling site (due to closed roads, flooded or low water levels) can be circumvented by finding a new access point, the inability to access a sampling site requires a staff member to use common sense. If the site is inaccessible due to unsafe conditions (e.g., waves), the FI will schedule a new day to return to the lake and finish the sample. At no point will the safety of the staff member(s) be compromised.

Table 7. Total Number of Samples to be Collected by Type During the 2021 Fish Tissue and Water Column Selenium and Mercury Project.

Sample Type	Samples per visit	Visits per year	Samples per year	Number of sites	Total QA samples	Total collected
Trace Metals, dissolved	1	30	1	30	3	33
Temperature, Dissolved Oxygen, pH, Specific Conductance	1	30	1	30	0	30
Secchi disk transparency	1	10	1	10	0	10

[1]Total number of duplicate and blank samples based on a QA sample collected on the first sample of the year then every tenth (i.e., 1, 10, 20, etc.)

B1.2 Sample Locations

Water chemistry and fish tissue will be collected from a total of 30 sampling locations in 2021. Twenty (20) sites will be visited from rivers and streams along with a subset of lakes/reservoirs listed in Table 1. Water quality data will be collected from the deepest area of the lake/reservoir and grab samples will be collected from rivers and streams. GPS readings will be recorded at each site, with accuracy of 10 meters. Using pre-selected sites, the NDDEQ increases comparability of data from that lake over time.

At the time of this publication, exact sampling locations are yet to be determined. For river/stream sites, confounding factors of current hydrology, landowner permission, safety, etc. will ultimately determine exact sampling locations. These factors will be evaluated during the site reconnaissance process which typically takes place in May. Also, uncertainty exists around whether lake/reservoir sampling will take place for the Fish Tissue and Water Column Selenium and Mercury Project in 2021 due to uncertainty of sampling equipment. Currently, a custom-built electrofishing boat is being built and is

due to arrive at the NDDEQ in late summer. Unforeseen circumstances (i.e., Covid-19, supply shortages, etc.) may prevent the prompt delivery of the electrofishing boat and, in this case, lake/reservoir sampling will be put on hold until the 2022 sampling season or the NDDEQ will coordinate with the NDGF Department and their fall surveys. This will depend on time, staff, and other resources available at the time.

B1.3 Sampling Frequency

One sampling visit will occur in June – early August for river and stream sites and one sampling visit will occur for the subset of lakes and reservoirs selected for sampling (Table 2).

B2. Sampling Methods

Table 8 provides a summary of project sampling methods (NDDEQ, draft 2020). Detailed descriptions of all field-sampling methods are described in Appendices A-C. Table 9 provides a fish species list associated with previous NDDEQ river and stream sampling locations in ecoregion 46, specifically the Souris and James River basins. Exact species presence and abundance is unknown; therefore, fish tissue sample collection efforts will be 'opportunistic' as it will depend on what species/abundances are present at the time of sampling.

Sampling equipment (e.g., churn splitters) will be cleaned using a phosphate-free soap and tap water and then air-dried. Water quality meters will be decontaminated in accordance with meter-specific requirements or suggestions (manual available at YSI). All byproducts can be washed down the drain.

Any problems encountered in the field during sampling will be documented in writing by the FI and given to the DPM for review and for corrective actions to be taken. The document will be updated when corrective action has been taken and describes the action taken. This document will be signed by the FI and DPM and filed and stored by the FI.

Table 8. Sampling Methods.

Matrix/ Substrate	Parameter	Sampling Equipment	Sampling Method	Max Holding Time	Sample Container	Sample Preservation and Care
Transparency	Secchi disk transparency	1	1	NA	NA	NA
Water column	Temperature, pH DO, and Specific conductance	2	2	NA	NA	NA
Water column	Chemistry	3	3	3	3	3
Fish tissue plugs	Fish tissue mercury	4	4	4	4	4
Whole fish tissue	Fish tissue selenium	5	5	5	5	5
Grab samples	Chemistry	6	6	6	6	6

1 - Appendix A 4 – Appendix D

2 - Appendix B 5 – Appendix E
3 - Appendix C 6 – Appendix F

Table 9. List of Potential Fish Species for River and Stream Sites Encountered in 2016

Species	Total Individuals	Density	% Occurrence	# of Sites	Trophic
Fathead minnow	1783	25.71%	80%	16	Omnivore
Sand Shiner	1585	22.86%	55%	11	Insectivore
Common shiner	1196	17.25%	30%	6	Insectivore
White sucker	805	11.61%	100%	20	Omnivore
Black Bullhead	680	9.81%	70%	14	Omnivore
Creek chub	190	2.74%	20%	4	Insectivore
Iowa Darter	121	1.75%	50%	10	Insectivore
Blacknose dace	114	1.64%	25%	5	Insectivore
Johnny darter	112	1.62%	35%	7	Insectivore
Common carp	77	1.11%	40%	8	Omnivore
Red Shiner	60	0.87%	10%	2	Insectivore
Blackside Darter	45	0.65%	30%	6	Insectivore
Northern pike	41	0.59%	45%	9	Piscivore
Tadpole madtom	36	0.52%	35%	7	Insectivore
Walleye	23	0.33%	10%	2	Piscivore
Yellow perch	19	0.27%	15%	3	Insectivore
Shorthead redhorse	15	0.22%	10%	2	Insectivore
Trout-Perch	15	0.22%	10%	2	Insectivore
Brook Stickleback	11	0.16%	20%	4	Insectivore
Orangespotted Sunfish	4	0.06%	10%	2	Insectivore
Bluegill	1	0.01%	5%	1	Insectivore
White crappie	1	0.01%	5%	1	Piscivore
Total_#_fish	6934	100.00%	Total_#_Sites	20	NA

B3. Sample Handling and Custody Requirements

Following sample collection in the field, all the chemical water samples will be hand delivered to the Division of Laboratory Services Chemistry laboratory in Bismarck, North Dakota. Holding times for analytes collected are listed in Table 7. Samples delivered to the lab will be temperature-checked upon arrival with a desired maximum temperature of 4°C. This temperature requirement, however, cannot always be met and can be dependent upon the time between sample collection and sample delivery. Samples collected that are preserved to 4°C will be kept on ice in an airtight cooler until they are delivered to the lab.

Fish tissue samples for selenium collected in rivers and streams will be handled in one of two ways. For small fish species (<200 mm), several individuals will be composited into one sample for selenium analysis (Task 2, pg 8). A minimum of 5 grams of composite

material will be placed into 20 mL conical bottom centrifuge tubes and frozen until a batch sample can be shipped to EPA Region 8 laboratory for analysis using selenium method 200.8 to be analyzed during the slower winter months. For fish species larger than 200 mm, pairs of individuals meeting the 75% rule will be composited into 20 mL centrifuge tubes. For example, if four white suckers are collected within the 75% rule, 2 samples of 2 individuals will be retained for composite tissue samples.

Regarding mercury analysis associated with rivers and streams, these samples will be collected opportunistically as species of interest are encountered. Species of interest include walleye, yellow perch, northern pike, bluegill, and crappie (white or black). As species of interest are encountered, muscle tissue plugs will be collected following Appendix D. Fish tissue mercury plugs will be hand delivered to the North Dakota Department of Environmental Quality lab with appropriate custody forms, etc.

Fish tissue selenium samples collected in lakes and reservoirs will follow the same protocols mentioned above. Smaller fish species (<200 mm) will have several individuals composited into one sample with 5 grams of composite material placed into 20 mL conical vials. Fish tissue mercury plugs will be collected from larger species of interest (walleye, yellow perch, northern pike, bluegill, and crappie) and, in addition to the tissue plugs, whole individuals will be retained for composite selenium to be submitted to EPA Region 8 lab. Fish tissue plugs for mercury analysis will be hand delivered to the NDDEQ lab in Bismarck with appropriate custody forms.

B4. Analytical Methods Requirements

All water samples will be analyzed according to methods and procedures described in the NDDEQ Division of Chemistry QAP (Table 4). For more information on equipment used, performance criteria, methods used, disposal procedures, or procedures when failure occurs, see the North Dakota Department of Environmental Quality Division of Chemistry Quality Assurance Plan (QAP) (revision March 2020).

All data should be received from the lab no later than November 30th, 2021. This will allow sufficient time for WMP staff to quality check the data before entering data into WQX and SID by January 15th, 2022.

B5. Quality Control

For this project, the FI will be responsible for taking field measurements and collecting water quality samples. Equipment used for field measurements will be calibrated according to the manufacturer's specifications immediately before and after each sampling trip. Furthermore, every tenth water quality sample will have a duplicate and a field bottle blank collected for chemical analysis. For information on lab QA procedures, see the lab's QAP (revision March 2020).

Control limits and corrective actions for laboratory analysis are outlined in the lab's QAP (revised 2020). For field analyses (i.e., in situ monitoring), probes are checked against

known calibration check solutions for pH and specific conductance. If probes do not meet DQOs (Table 5), probes will be re-calibrated and re-checked. There may be other reasons that probes do not meet DQOs (e.g., calibration check has been opened for too long), but if a probe begins to "drift" excessively (i.e., bounces around and does not stabilize) it is likely that the result needs to be flagged (or not recorded) and the probe needs to be replaced. Upon return to the office, the sonde will be taken out of service until the probe is replaced.

Since this is a new project for primary data collection, data will not be checked for 90th and 10th percentiles of historical data. Rather, as a long-term dataset will be compiled. Once 5-10 years of data are compiled, then future data collection will be compared expected values. Data will, however, be checked for accuracy by evaluating blank data for "hits".

B6. Instrument/Equipment Testing, Inspection and Maintenance

It is the FI's responsibility to test, inspect and maintain all field instruments and equipment. Field equipment will be inspected and tested prior to sampling activities to ensure that the equipment is functioning properly and to allow time for replacement or repair of identified deficiencies. Prior to sampling and following calibration of the water quality meter, probes will be checked using known conductivity and pH checks. Values will be compared to DQOs and issues will be remediated, as needed. All field equipment will be maintained according to the manufacturer's specifications. After the sampling season, YSI meters will be submitted for winter maintenance annually to check each meter for defects and replace probes, if necessary. Spare parts for water quality sampling equipment and water quality sondes are stored at either the Gold Seal Building or Environmental Training Center. The laboratory is responsible for laboratory equipment failure.

Periodic maintenance for lab equipment is outlined in the QAP (revised 2020).

B7. Instrument/Equipment Calibration and Frequency

Instrument and equipment (i.e., temperature and DO, pH and specific conductance meters) will be calibrated according to the manufacturer's specifications (meter-specific manuals available at YSI) and at the frequency outlined in the SOP (Appendix B). For information regarding the lab's calibration procedures, please reference their QAP (revision 2020).

B8. Inspection/Acceptance of Supplies and Equipment

Careful and thorough planning is necessary to ensure the efficient completion of the field sample collection tasks. A general checklist of field equipment and supplies is provided in the description of the SOPs (Appendices A - C). It is the responsibility of the FI to gather the necessary sampling supplies and equipment prior to each sampling trip. Bottles are provided by the laboratory and are stored prior to each trip. Other consumables are

ordered by the DPM prior to each sampling season, if needed, and are stored in the Environmental Training Center or equipment garage.

The laboratory is responsible for the inspection of and acceptable activities associated with lab testing supplies and consumables.

B9. Data Management

The Fish Tissue and Water Column Selenium and Mercury project does not utilize data from other agencies. Samples will be documented and tracked through sample identification labels, field and laboratory recording forms and sample identification/custody forms. Fish tissue plugs/composites collected for chemical analysis will be shipped to region 8 EPA lab and the water samples will be hand-delivered to the Division of Laboratory Services laboratory in Bismarck, ND by the FI.

Results of the chemical analysis of water samples are transmitted from the Division of Laboratory Services to the WMP Program Manager via hard copy report electronically as an ASCII text file. The lab's QAP (revised 2020) provides an outline of how the data are processed from the lab and outlines their quality assurance processes. At least 5-10% of all sample results will be reviewed by the DPM prior to entry in the SID database. Prior to entry into SID, data will be transferred to a temporary database where the FI will 1) compare duplicate and sample data for QA samples, if available, 2) evaluate blank samples for any "hits", and 3) compare data collected to historical data to ensure data are within limits of historical data (see Table 6). Following data validation, results are then transmitted electronically and stored by the Division of Water Quality's WMP in SID. Field measurement data (e.g., Secchi disk transparency, temperature, DO concentration, pH and specific conductance) are directly entered into SID by WMP staff. Data validation will be ensured by the FI via successful calibration of water quality meter and that checks of parameters were within the range for precision and accuracy. If calibration checks are outside of these ranges, data will still be entered into SID, but will be flagged in the database. Additionally, all data files from EPA R8 laboratory will be transmitted to the WMP Program Manager along with the WMP Database Coordinator (Figure 1).

After review by the WMP Program Manager, sample results will be retained by the DPM for data reduction and analysis. All results are then entered into EPA's WQX.

All revisions of this SOP will be archived on the Y drive as a method of document control to show change over time. The revision page at the beginning of the document also provides a short summary of changes made with each revision.

C. Assessment and Oversight

C1. Assessment and Response Actions

The project will be assessed following each sampling date range (e.g., the project will be assessed in August 2021 following June sampling to allow for receipt of all data from the

lab). This assessment will determine if all samples were collected in conjunction with QAPP data needs. This assessment will also describe issues encountered when sampling, if any, and how these issues were reconciled. This assessment will be documented in a short report (about one page) and sent to DPM and QAC. Four assessments will be completed (coinciding with four sampling runs) with a virtual copy saved to the Y drive and a hard copy filed by the FI in the project folder.

Assessment activities and corrective actions have been identified to ensure that sample collection activities are conducted as prescribed and that the measurement quality objectives and data quality objectives established by this QAPP are met. The QA program under which this project will operate includes performance and system audits with independent checks of the data obtained from sampling activities. Either type of audit could indicate the need for corrective action. The essential steps in the program are as follows:

- Identify and define the problem;
- Assign responsibility for investigating the problem;
- Investigate and determine the cause of the problem;
- Assign and accept responsibility for implementing appropriate corrective action;
- Establish effectiveness of and implement the corrective action; and
- Verify that the corrective action has eliminated the problem.

Immediate corrective actions are: (1) part of normal operating procedures, (2) noted on project field and laboratory recording form and (3) the responsibility of the DPM or FI. Problems not solved this way may require more formalized long-term corrective action. In the event that quality problems requiring attention are identified, the DPM will determine whether attainment of acceptable data quality requires either short- or long-term actions. Failures in the chemical analysis system (e.g., performance requirements are not met) and corrective actions for those failures are beyond the scope of this QAPP.

Communication and oversight will proceed from the FI to the DPM. The DPM will be available throughout the entire sampling period to address questions and receive communications of sampling status from the FI.

The FI will initiate corrective actions when a problem is identified and note the problem and corrective action in his logbook. In the event that the problem cannot be corrected immediately, the FI will contact the DPM to determine the best way to rectify the problem and obtain accurate and useable data. When corrective actions have been taken and a sufficient time period has elapsed that allows a response, the response will be compared with project goals by the DPM. The DPM will verify that the corrective action has been appropriately addressed to eliminate the problem. The DPM has the authority to

stop work on the project if problems affecting data quality are identified that will require extensive effort to resolve. When the FI contacts the DPM with a problem, the FI will make a record of the problem and the corrective action taken.

Performance audits are qualitative checks on different segments of project activities, and are most appropriate for field sampling and laboratory analysis activities. A field audit of field sampling activities will be conducted at least once during the project. This audit will be conducted early during the project field season in case any problems are identified they can be corrected quickly to minimize the possibility of compromising data. Field audit techniques include checks on sampling equipment and the review of sampling methods.
System audits are qualitative reviews of project activity to check that overall project quality is functioning and that the appropriate QC measures identified in the QAPP are being implemented. The DPM will conduct periodic internal system audits during the project.

C2. Reports to Management

Problems and corrective actions identified by the FI will be reported to the DPM each week during the field season, or as necessary. Significant problems identified by the FI as well as problems and corrective actions identified by the DPM during the field audit will be reported to the Division of Water Quality Director and the EHS QAC.

D. Data Validation and Usability

D1. Data Review, Validation and Verification Requirements

Data review and validation services provide a method for determining the usability and limitations of data, and provide a standardized data quality assessment. The FI and the DPM will review all field and laboratory report forms, while all sample custody forms for chemical analysis will be reviewed by the DPM for completeness and correctness. The FI will be responsible for reviewing all data entries and transmittals for completeness and adherence to QA requirements. Data quality will be assessed by comparing entered data to original data or by comparing results with the measurement performance criteria summarized in Section A4.2 to determine whether to accept, reject, or qualify the data. Results of the review and validation processes will be reported to the DPM.

D2. Verification and Validation Methods

The DPM will review all field and laboratory record forms. The DPM will review a minimum of five percent of field and laboratory record forms and all of the sample custody forms for chemical analysis. Any discrepancies in the records will be reconciled with the field personnel and recorded in the logbook. Meter-specific calibration logs are kept with each meter. When completing the log, the FI will include their initials and record all information regarding the calibration and quality checks of the probes. Checklists and forms used in the field are available in the appendices.

The submission of samples to the Division of Laboratory Services-Chemistry laboratory will include a Sample Identification/Custody Record sheet (Appendix C,D,E) documenting the site location, sampling date and time. The Division of Laboratory Services-Chemistry laboratory to ensure that holding times have not been exceeded will check this information. The laboratory will report violations of holding times to the DPM. The DPM, in consultation with the FI and Division of Laboratory Services-Chemistry personnel, will determine whether to proceed with the analysis of that sample and/or analyte.

D3. Reconciliation with Data Quality Objectives and User Requirements

As soon as possible after each sampling event or the analysis of each sample, calculations and determinations for precision, completeness, and accuracy will be made by the FI and compared to the criteria discussed previously. This will represent the final determination of whether the data collected are of the correct type, quantity, and quality to support their intended use for this project. Any problems in meeting the performance criteria (or uncertainties and limitations in the use of the data) will be discussed with the DPM, and will be reconciled, if possible. These could arise from QA samples not meeting DQOs or samples being outside of the range of values given in Table 6. These data will be considered further and, if necessary, will be flagged and commented to communicate the uncertainty from either "hits" in blanks (i.e., potentially adding parameter to the sample) or deviation using duplicates (i.e., concern over the precision of that set of data). Uncertainty arising from calibration and subsequent checks will be communicated by flagging and commenting for each reading of that parameter (e.g., pH). It is the responsibility of the FI to compile questionable data and present it to the DPM for further consideration. If reconciliation cannot be completed, however, it will be at the discretion of the DPM whether data will be rejected and not distributed in SID.

Literature Cited

Clesceri, L. S., A. E. Greenberg, A. D. Eaton (eds.). 2017. Standard Methods for the Examination of Water and Wastewater, 23rd Edition. American Public Health Association, American Water Works Association, and Water Environment Federation, Washington, D. C.

EPA. 2001 (reissued May 2006). EPA Requirements for Quality Assurance Project Plans. U.S. Environmental Protection Agency, Quality Assurance Division, Washington, D.C. EPA/QA/R-5.

EPA. In revision. Quality Assurance Project Plan for Monitoring for Harmful Algal Blooms. US EPA Region 8 Water Division, Denver, CO.

NDDEQ. 2019. Standards of Water Quality for the State of North Dakota. Chapter 33-16-02 of the North Dakota Century Code. North Dakota Department of Health, Division of Water Quality, Bismarck, ND.

NDDEQ. March 2020. North Dakota State Department of Health Laboratory Services Division Chemistry Laboratory Quality Assurance Program. North Dakota Department of Environmental Quality, Division of Laboratory Services, Bismarck, ND.

NDDEQ. December 2020. Quality Assurance Program Plan for Water Quality and Watershed Projects/Studies. Division of Water Quality, North Dakota Department of Environmental Quality, Bismarck, ND.

NDDoH. April 2019. North Dakota 2018 Integrated Section 305(b) Water Quality Assessment Report and Section 303(d) List of Waters Needing Total Maximum Daily Loads. North Dakota Department of Health, Environmental Health Section, Bismarck, ND.

NDDoH. March 2016. Quality Management Plan for the North Dakota Department of Health, Environmental Health Section, Bismarck, ND.

NDDoH. March 2011. Standard Operating Procedures for Field Samplers. North Dakota Department of Health. Bismarck, ND.

APPENDIX A

STANDARD OPERATING PROCEDURES FOR MEASURING SECCHI DISK TRANSPARENCY

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	Joe Nett	
Program Manager	Aaron Larsen	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Measuring Secchi Disk Transparency
Type: Standard Operation Procedure #X
Version: 1.0
Date: 03/29/2019
Author: Joe Nett

REVISION HISTORY

Revision	Change Description	Date	Authorization
0	Document Creation/Control	03/29/2019	

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 2

4.0 CAUTIONS 3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES 3

7.0 EQUIPMENT AND SUPPLIES 3

8.0 PROCEDURE 3

9.0 DATA AND RECORD MANAGEMENT 4

10.0 QUALITY ASSURANCE AND QUALITY CONTROL 4

11.00 REFERENCES 4

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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 performing Secchi disk measurements (also referred to as Secchi readings or Secchi depths) in lakes, reservoirs, and wetlands. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers. Secchi disk readings are essentially a measure of transparency and give an indication of water clarity. The DWQ uses this data in several ways. As an important indicator for lake water quality assessment, Secchi readings can allude to algae and/or suspended sediment concentrations. Secchi depth values are one component used to calculate the Carlson Trophic State Index, a measure of the degree of eutrophication in a lake/reservoir. The depth of the Secchi reading can also be used to determine the depth at which surface water samples are to be collected. There is no one standard technique for performing Secchi readings and there is ongoing debate in the scientific community regarding whether Secchi readings should be taken in the shade vs. sun, with the naked eye vs. a viewing box, at the point of disk disappearance vs. reappearance vs. an average of the two, with an all white disk vs. a black and white disk, etc. (Smith, 2000). Therefore, the best way to make data comparable for a monitoring project/program is to choose one procedure and have all data collectors use that procedure consistently. The procedure below was chosen by DWQ with a goal of reducing variability due to glare and degree of cloud cover.

2.0 SUMMARY OF METHOD

The Secchi disk is lowered into the water until it disappears from view. The depth at which the Secchi disk reappears after vanishing is the recorded Secchi reading. The sampler takes the reading on the shady side of the boat without wearing sunglasses.

3.0 HEALTH AND SAFETY WARNING

Field personnel should take appropriate precautions when operating watercraft and working on, in, or around water. All boats should be equipped with safety equipment such as personal flotation devices (PFD's), oars, air horn, etc. 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

Watercraft must be stationary while performing Secchi readings. A moving watercraft will produce invalid readings because the Secchi disk will not be aligned vertically in the water column. An additional anchor may be needed to further secure watercraft. Extreme wave action may also produce invalid readings. In these cases, round the Secchi reading to the nearest 0.1 meter as best as possible, making sure to note the field conditions on a field sheet or in a field notebook.

5.0 INTERFERENCES

Several factors may affect the Secchi reading. Since the eyesight of samplers may vary, all readings on the same waterbody ideally should come from the same person. Weather conditions and site conditions (e.g. overcast skies, water surface scum, dark colored water, etc.) should be recorded so that outlier readings may be explained.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel taking Secchi readings 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

A Secchi disk is made out of a 20-cm-diameter Plexiglass disk painted with four alternating black and white quadrants. This disk is attached to a metered tape by a series of nuts and bolts. The tape is marked off in meters (subdivided by tenths of meters). Before use, make sure the markings on the tape are still clearly visible.

_____ Copy of this SOP
_____ Secchi disk and metered tape
_____ Field forms, pencils or pens

8.0 PROCEDURE

Upon arrival to the sample site, establish which sampler is going to perform the Secchi reading(s).

1) Retrieve the Secchi disk from storage.

2) Move to the shady side of the boat and wait for the boat to be as stationary as possible before lowering the Secchi disk.

3) With sunglasses off, lower the disk slowly; make sure the tape is straight up and down.

4) Lower the Secchi disk to the point of vanishing and slowly raise it back up until it reappears. Move the disk up and down until the exact vanishing/reappearing point is found. At this point, read the tape where it is entering the water; this is the Secchi reading. One can visually read the tape or use your hand to mark the tape where it meets the water's surface.

5) Pull the disk out of the water and record the tape measurement to the nearest 0.1 meter.

9.0 DATA AND RECORDS MANAGEMENT

Secchi readings will be recorded on the field form (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 reading such as high winds/wave action. Field forms and notes should be stored in the appropriate project folder at DWQ.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

There are limited Quality Assurance and Quality Control (QA/QC) procedures for Secchi readings. Duplicate readings may be performed at sites where duplicates samples are to be collected or two readings may be averaged by the sampler, if desired. For quality control, Secchi readings should ideally be taken by one person for an entire sampling trip. For long-term monitoring stations, all readings at the same location should ideally be taken by one person. A project-specific Sampling and Analysis Plan (SAP) may require additional quality control activities, for example having two samplers each record Secchi readings in order to measure variability between samplers.

11.0 REFERENCES

Smith, D.G. 2000. Standardization of secchi disk measurements, including use of a viewer box. Proceedings of the National Water Quality Monitoring Conference 2000: Monitoring for the Millennium, Austin, TX. National Water Quality Monitoring Council.

Related DWQ SOPs

Standard Operating Procedures for the Measurement of Temperature and Dissolved Oxygen Profiles in Lake and Reservoirs

Standard Operating Procedures for the Collection of Lake Water Quality Samples

APPENDIX A

Field Reporting Form

Lake Profile Record Field Log
North Dakota Department of Environmental Quality
Division of Water Quality
Watershed Management Program

Project Code:		Project Name:	
Site Identification:		Sampler(s):	
Site Description:			
Date:	Time:	Ambient Temp:	Wind Speed: (mph)
Wind Direction:	% Cloud Cover:	Secchi Disk: (m)	Baro: (mm/Hg)
Chlorophyll-a:	Phytoplankton:	Initial DO:	Final DO:

Sample Depths: _____ Meters, _____ Meters, _____ Meters, _____ Meters

Comments:

Depth (m)	Temp (c)	DO (Mg/L)	pH	Specific Conduct.	Comments

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."

Printed Name	Signature	Date

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature

APPENDIX B

STANDARD OPERATING PROCEDURES FOR TAKING FIELD MEASUREMENTS IN STREAMS, RIVERS, LAKES AND WETLANDS USING HAND HELD METER(S)

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	McKenzie Schick	
Program Manager	Aaron Larsen	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Taking Field Measurements in Streams, Rivers, Lakes, and Wetlands Using Handheld Meter(s)
Type: Standard Operation Procedure #7.02
Version: 3.0
Date: 01/08/2020
Author: McKenzie Schick

REVISION HISTORY

Revision	Change Description	Date	Authorization
0	Document Creation/Control	01/08/2020	

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 2

4.0 CAUTIONS.....3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES.....3

7.0 EQUIPMENT AND SUPPLIES 4

8.0 PROCEDURE 4

9.0 DATA AND RECORD MANAGEMENT 5

10.0 QUALITY ASSURANCE AND QUALITY CONTROL 6

11.0 REFERENCES.....6

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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 performing field measurements using a handheld meter in rivers, streams, lakes, and wetlands. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers. The following is summarized from the Field Guide for Collecting and Processing Stream Water Samples for the National Water Quality Assessment Program (Shelton 1994).

Measurements of pH can provide some of the most important limnological information pertaining to a water-body. The pH of a solution is a measure of the effective hydrogen-ion concentration. pH is in logarithmic units using a scale from 1 to 14. Water bodies with a pH of less than 7 are considered acidic, while water bodies with a pH greater than 7 are considered basic or alkaline.

Dissolved gases such as carbon dioxide, hydrogen sulfide, and ammonia appreciably affect pH. Due to this, pH must be taken in the field, as a significant change can occur within several hours or even minutes after sample collection.

Specific conductance is the reciprocal of resistance in ohms and is a measure of the capacity of water or another substance to conduct an electrical current. Specific conductance is reported in micro siemens per centimeter at 25 degrees Celsius. The specific conductance of water is determined by the types and quantities of dissolved substances in the water. Thus, specific conductance indicates the concentrations of dissolved solids in water.

The specific conductance of water may change significantly with time because of pollution, precipitation, absorption, ion exchange, oxidation, or reduction. Therefore, specific conductance should be measured in the field.

Temperature and dissolved oxygen (DO) measurements may also provide some of the most important limnological characteristics of a water body (e.g., biological and biochemical reactions). Therefore, water temperature and DO should be measured in the field.

2.0 SUMMARY OF METHODS

The handheld meter is calibrated daily, or prior to each use, using manufacturer's instructions. The calibration is logged into a calibration log (Appendix A). The probes are then lowered into the water and a measurement for temperature, DO, pH, and specific conductance is recorded.

3.0 HEALTH AND SAFETY WARNING

Field personnel should take appropriate precautions when operating watercraft and working on, in, or around water. All boats should be equipped with safety equipment such as personal flotation devices (PFD's), oars, air horn, etc. 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.

Field personnel should also be aware of wildlife, insects, and plants that could be harmful as well as heat stroke and hypothermia. A first aid kit should be accessible for any potential cuts, stings, bites, or contact with poisonous plants. Ensure there is access to water, sunscreen, insect repellant, and extra clothing.

4.0 CAUTIONS

- Watch for bubbles or wrinkles in the membrane, these will cause errors in the DO reading (membrane handheld meters).
- Calibration solutions need to be stored properly. (e.g. room temperature and not in vehicles exposed to temperature extremes).
- Use calibration checks to ensure probes are working correctly.
- Confirm guard is properly installed for every sample.
- Probes must be checked before each sampling season to ensure that they are in working condition. Send to YSI for maintenance if necessary.
- Do not use expired probes.
- Calibrate specific conductance first.
- Take surface readings in lakes prior to sample collection.
- Take readings in rivers and streams prior to sample collection or upstream of sample collection.
- If traveling and staying overnight, handheld meters should not be left in vehicles overnight and should be stored at room temperature when possible.

5.0 INTERFERENCES

Membrane Handheld Meters Only: The membrane of a DO electrode is permeable to additional gases other than oxygen, such as hydrogen sulfide (H_2S). Caution should be taken when using the membrane electrode in low DO waters since the presence of H_2S may lower the cell sensitivity. This interference can be reduced by frequently changing the membrane and calibrating the electrode. For optic DO probes, make sure that the sensor is clear of film and if a wiper is present, replace the wiper if dirty.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel taking field measurements using a handheld meter 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 personnel 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

- ☐ Handheld meter(s)
- ☐ Maintenance kit (Potassium chloride/KCl solution (membrane meters only), spare membranes (membrane meters only), batteries, battery charger)
- ☐ Site Map
- ☐ Bathymetric lake map for lake sampling, if available
- ☐ Field report form
- ☐ Pens or pencils
- ☐ pH 7 and pH 10 calibrating buffer
- ☐ Specific conductance 1,413 µS cm^{-1} calibration solution
- ☐ pH 9 calibration check
- ☐ Specific conductance 1,008 µS cm^{-1} calibration check
- ☐ Power ice auger (winter sampling)
- ☐ Ice skimmer (winter sampling)
- ☐ Meter stick (winter sampling)
- ☐ Sled (winter sampling)
- ☐ Personal Flotation Device
- ☐ Boat or canoe, if needed

8.0 PROCEDURE

Streams and Rivers

1. Calibrate the meter using a solution with known specific conductance (1,413 µS cm^{-1} preferred), pH buffer solutions of 7.0 and 10.0, and calibrate dissolved oxygen according to barometric pressure following manufacturer's instructions.

2. Confirm calibration effectiveness using calibration check solutions.

3. Record calibration information in the equipment calibration log (Appendix A). Note: Calibration logs are meter specific.

4. Locate the main current of the stream or river. Note: When drilling a hole through the ice, be sure not to disturb the sediment with undue agitation.

5. Place the guard over the exposed probes and lower the probe to that depth which is approximately 60% the total water depth below the surface. For example, if the stream is five feet deep, take the measurement three feet below the surface.

6. Read temperature, dissolved oxygen, pH, and specific conductance if using a multi-probe meter and record. If using a single probe meter wait for the temperature reading to stabilize (30 seconds minimum), record the temperature reading on the

stream and river sampling field log (Appendix A), switch the display to read dissolved oxygen, allow the dissolved oxygen reading to stabilize and record the dissolved oxygen concentration on the field report form. NOTE: To achieve an accurate reading some units require a stirring unit or for the sampler to gently move the probe up and down two to three inches to circulate water across the membrane (membrane handheld meters only).

Lakes and Wetlands

1. Locate the desired sampling location and anchor boat or drill a hole through ice. Note: When drilling a hole through the ice do not disturb the water column with undue agitation.

2. Calibrate to manufacturer's instructions and record calibration information in the equipment calibration log (meter specific).

3. Fill in the station identification information on the field report form. Also, measure and record ice thickness and snow depth in the comments section (winter sampling) (Appendix A).

4. Remove the storage cup and replace it with a protective guard. Lower the probe to 0.5 meters depth, or just below the ice.

5. Read the temperature, DO, specific conductance, and pH concentrations and record on lake or wetland sample record form (Appendix A). If using a single probe meter wait for the temperature reading to stabilize (30 seconds minimum), record the temperature reading on the field report form, switch the display to read dissolved oxygen, allow the dissolved oxygen reading to stabilize and record the dissolved oxygen concentration on the field report form. NOTE: To achieve an accurate reading some units require a stirring unit or for the sampler to gently move the probe up and down two to three inches to circulate water across the membrane (membrane handheld meters only).

6. Lower the probe to the next depth interval and repeat step 5. Readings should be taken at every meter (i.e. 0.5, 1, 2, 3…) if greater than three meters deep. Every half meter (i.e. 0.5, 1, 1.5, 2, 2.5…) if three meters depth or less.

7. Repeat step 6 until 0.5 meters of the bottom.

8. Retrieve probe from bottom of water body, rinse thoroughly and replace the storage cup. Recheck the surface reading to ensure the reading is within 0.2 mg L^{-1} of initial reading, following profile measurements to ensure the precision and accuracy of the measurements. If the reading is not within 0.2 mg L^{-1}, re-calibrate the meter and measure the profile again, repeating steps 5 through 7.

9.0 DATA AND RECORDS MANAGEMENT

Handheld meter measurements will be recorded on the field form (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 reading such as high winds/wave action, cattle in water, observed flow, water surface, water clarity, water color, water odor, visual algae cover, number of dead fish, present weather, estimated inches of rain fall in past 72 hours, and any comments. Field forms and notes should be stored in the appropriate project folder at DWQ.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

The meter(s) should be calibrated before sampling trip following the manufacturer's instructions and the calibrations should be recorded. Additionally, temperature probes will be checked annually against a certified NIST thermometer.

11.0 ANS DECONTAMINATION

In waters that have been classified as ANS infested, the meter must me decontaminated (Decon). To decon the meter pH 4.00 solution will be used. Triple rinse probes using pH 4.00 solution and then store the probes in pH 4.00 solution.

11.0 REFERENCES

Gibs, Jacob, et al. "Use of Multiparameter Instruments for Routine Field Measurements." *USGS*, USGS, Mar. 2012, https://pubs.usgs.gov/twri/twri9a6/twri9a68/twri9a6_6.8.pdf.

Shelton, Larry R. "Field Guide for Collecting and Processing Stream-Water Samples for the National Water-Quality Assessment Program." *USGS*, USGS, 1994, https://pubs.usgs.gov/of/1994/0455/report.pdf.

APPENDIX A

Field Reporting Forms

River and Stream Sampling Field Log
North Dakota Department of Environmental Quality
Division of Water Quality - Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	

Lake Profile Field Log
North Dakota Department of Environmental Quality
Division of Water Quality – Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Project Code:		Project Name:	
Site Identification:		Sampler(s):	
Site Description:			
Date: / /	Time: :	Ambient Temp:	Wind Speed: (mph)
Wind Direction:	% Cloud Cover:	Secchi Disk: (m)	Baro: (mm/Hg)
Chlorophyll-a:	Phytoplankton:	Initial DO:	Final DO:
Sample Depths: _____ Meters, _____ Meters, _____ Meters, _____ Meters			
Comments:			

Depth (m)	Temp (c)	DO (Mg/L)	pH	Specific Conduct.	Comments

Depth (m)	Temp (c)	DO (Mg/L)	pH	Specific Conduct.	Comments

Wadable Wetland Field Sampling Form
North Dakota Department of Environmental Quality
Division of Water Quality – Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Site ID:	Wetland Name or description:			County:
Observer:			Date:	Time:
Aquatic Zone Description:				
Ambient Temp:	Wind Speed:		Wind Direction:	
Cloud Cover % of 100	Secchi (m)	chlorophyll-a=(y / n)		Phytoplankton=(y / n)
Comments:				
Water Chemistry Taken At	Meters,	Meters,	Meters,	Meters,

Depth (m) or Location	Temp. (C)	D.O. (mg/L)	pH	Conductivity (umhos/cm)	Comments

Site Drawing:

NORTH Dakota | Environmental Quality
Be Legendary.™

Mutiple Sample Set
Sheet #_____of_____

Sample Arrival Time-Stamp:_____

CUSTODY RECORD AND ANALYSIS REQUEST – Watershed Management Program

Account #	**Project Code:**	**Project Name:**	**FOR LABORATORY USE ONLY** **Nutrient/Nitrate bottle(s) checked for preservation by:** **Temp of Cooler:**
DEQ Program:	**DEQ Project #:**	**DEQ Cost Center #:**	**Point of Contact/DPM:**
Sampled By:		**Sampler Phone #:**	
Analysis Requested:		***Collection Method: (See Note)**	**Matrix: Soil Water Other (explain)** _____ **Enforcement? Yes No**

Lab ID (Enter # from lids of samples here)	Site ID/STORET #	Sample Location (Lat Long or TRS)	Sample Date	Sample Time	# of Bottles	Cooler #	Co-located Site ID and/or Comments	Depth in meters	Field Measurements	
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH

*** Collection Methods (Record Above):** Depth Integrated (DI) ~ Depth/Width Integrated (DWI) ~ Grab ~ 0-2 meter column
When collecting lake samples, you ***MUST*** include the sampling depth(s).

Relinquished by	Date and Time	Received by	Date and Time

Revised 6/15/20

ED_013266B_00000036-00063

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."

Printed Name	Signature	Date

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature

APPENDIX C

STANDARD OPERATING PROCEDURES
COLLECTING AND PRESERVING LAKE WATER QUALITY SAMPLES

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	Joe Nett	
Program Manager	Aaron Larsen	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Collecting and Preserving Lake Water Quality Samples
Type: Standard Operation Procedure #X
Version: 1.0
Date: 02/10/2021
Author: Joe Nett

REVISION HISTORY

Revision	Change Description	Date	Authorization

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 2

4.0 CAUTIONS.....3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES.....3

7.0 EQUIPMENT AND SUPPLIES 3

8.0 PROCEDURE 3

9.0 DATA AND RECORD MANAGEMENT 4

10.0 QUALITY ASSURANCE AND QUALITY CONTROL 4

11.00 REFERENCES.....4

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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 preserving water quality samples in lakes and reservoirs. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers.

2.0 SUMMARY OF METHOD

Water column samples should be reflective of the whole lake. To be representative of the lake, samples must be carefully collected, properly preserved and appropriately analyzed. The chosen method should align with the goals of the sampling project. For example, lake projects focused on assessing trophic state could choose to take only surface samples using a two-meter column sampler from the deepest point in the lake. If a project manager is focused on a nutrient budget or assessing mass balance, taking two to four samples from various depths from the deepest area of the lake may be the desired method. Information regarding sample bottles and preservation are available in the WMP's programmatic QAPP (NDDEQ, 2020)

3.0 HEALTH AND SAFETY WARNING

Field personnel should take appropriate precautions when operating watercraft and working on, in, or around water. All boats should be equipped with safety equipment such as personal flotation devices (PFD's), oars, air horn, etc. 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

When taking samples from near the water's surface, the sampler must ensure that there have been no re-suspended sediments (e.g., from dropping the anchor). If so, the sampler can either wait until the suspended sediment disappears from the photic zone or move the boat a short distance to an undisturbed area.

If the sampler disturbs the bottom sediments while collecting a sample (either collecting a bottom sample in a deep lake or a surface sample in a shallow lake), the sampler should adequately rinse out the sampling equipment and move a short distance to an undisturbed area and try again.

5.0 INTERFERENCES

Addressed in the previous section, samplers need to be aware of depth and suspended sediments.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel taking water quality samples from lakes and reservoirs 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

Supplies needed when collecting samples using a two-meter column sampler:

_____ Two-meter depth integrated column sampler (link for instructions)
_____ Two-gallon churn splitter
_____ Sample containers
_____ Acid for sample preservation (sulfuric and nitric)
_____ Labels
_____ Field forms
_____ Pens and/or pencils
_____ Packing tape to hold labels on bottles
_____ Cooler with ice or frozen gel ice packs
_____ Deionized (DI) water for sample blanks and decontamination
_____ For nutrient analysis using vacuum method
    _____ Vacuum filter holder

_____ Vacuum pump
_____ 0.45 µm membrane filters (Millipore HAWP 047 00 or equivalent)
_____ Pre-filters (Millipore AP40 0047 05 or equivalent)
_____ Forceps

_____ For nutrient analysis using peristalic method
_____ Power Drive (Compact Cat no. P-07533-50 or equivalent)
_____ Peristalic head (Easy Load II Cat No. P-77200-62 or equivalent)
_____ Inline 0.45 µm cartridge filters (Geotech dispos-a-filter or equivalent)
_____ Inline 5.0 µm cartridge filters (Geotech dispos-a-filter or equivalent), if necessary
_____ Tubing (Masterflex silicone Cat No. P-96400-24 or equivalent)

_____ For chlorophyll-a and -b analysis using vacuum method
_____ Vacuum filter holder
_____ Vacuum pump
_____ One 50-ml plastic vial for each chlorophyll filtration
_____ 0.65 µm glass fiber filters (Pall Corp. Glass Fiber Filters No. 61631, 0.47mm or equivalent)
_____ 500 ml graduated cylinder
_____ DI water for rinsing and decontamination
_____ Aluminum foil
_____ Forceps

_____ For phytoplankton sample collection
_____ Lugol's Solution (Add catalog information here)

8.0 PROCEDURE

Upon arrival to the sample site, establish which sampler is going to collect the water quality, chlorophyll and/or phytoplankton sample(s).

8.1 Sample collection:

1) Retrieve the sampler from storage.

2) When using a Kemmerer or Van Dorn sampler at discrete depths, for lakes and wetlands four meters deep or less that are not thermally stratified collect one sample at the one-meter depth interval.

3) When using a Kemmerer or Van Dorn sampler at discrete depths, for lakes and wetlands four meters deep or less that are thermally stratified or lakes greater than four meters deep that are not thermally stratified, collect one sample at the one-meter depth interval and one sample one meter off the bottom in the hypolimnion.

4) When using a Kemmerer or Van Dorn sampler at discrete depths, for lakes and reservoirs greater than four meters deep that are thermally stratified, collect one sample at the one-meter depth interval, one sample in the metalimnion (identified from the temperature profile recorded at the site) and one sample one meter off the bottom in the hypolimnion.

5) If using a two-meter column sampler instead, only one sample needs to be collected from the top two meters of water.

6) Complete sample labels with sample-specific information. Label all sample containers and place packing tape over the label to hold down the label. For further sample identification, consider writing sample information in the event of a lost or damaged label (e.g., sample number ["1" if first sample] and analysis type ["trace met" for Trace Metals]).

7) Lower the sampler into the water and pull through the water to properly rinse sampling equipment.

8) Collect samples beginning at the one-meter depth interval and progressing down the water column. Triple-rinse the churn splitter using lake water prior to filling the bucket completely.

9) Triple-rinse sample bottles for unfiltered samples (e.g., Nutrients Complete; Cations/Anions). Fill containers and preserve in accordance to guidance printed on the sample label or in the programmatic Quality Assurance Project Plan (QAPP). Sample filtration will be described below.

10) Place the filled, preserved samples in a cooler on ice.

11) Fill out the field report form (Reference), Sample ID/Custody Record (Reference) and the water column chemistry sample log (Reference).

12) Return churn splitter to vehicle immediately for sample filtration.

8.2 Field bottle blank sample collection:

1) Field blanks are collected with the first and every tenth sample (i.e., 1, 10, 20, etc.).

2) Label each sample container appropriately. **Note:** Field bottle blank samples are identified with STORET number 389990.

3) Triple rinse each bottle with DI water, except for your dissolved sample(s).

4) Fill each bottle with DI water, except for your dissolved sample(s).

5) Filter dissolved samples with DI water in accordance to methods discussed below.

6) Preserve each sample appropriately. **Do not** preserve dissolved nutrients until after filtering.

7) Place the sample in a cooler on ice.

8.3 Field duplicate sample collection:

1) Field duplicates are collected with the first and every tenth sample (i.e., 1, 10, 20, etc.). These samples are usually collected in conjunction with blank samples.

2) Collect a separate (or duplicate) sample in accordance to instructions above.

3) Place a label on each sample container. **Note:** Field duplicate samples should be identified with STORET number 389999.

8.4 Filtering nutrient samples using vacuum method:

1) Unpreserved dissolved nutrients samples should be filtered as close to collection as possible.

2) Put on new latex or nitrile gloves.

3) Rinse filter holder with DI water and re-assemble.

4) Load a pre-filter in the filter apparatus and connect the vacuum pump.

5) Leach the filter twice with approximately 250 ml of DI water each time (total of 500 ml).

6) Filter the sample through the pre-filter. Place the filtered sample back into a DI-rinsed 500 ml sample container.

7) Remove the pre-filter from the filter apparatus and repeat Step 3.

8) Load a 0.45 µm into the filter apparatus and connect the vacuum pump.

9) Repeat Step 5.

10) Filter the sample water through the 0.45 µm filter.

11) Triple-rinse the sample container with DI water and discard the rinse.

12) Transfer the filtered sample back into the rinsed sample container.

13) Preserve the sample with 2 ml 1/5 sulfuric acid or 0.2 ml concentrated sulfuric acid.

14) Place the preserved sample in the cooler on ice.

15) If additional samples require filtration, repeat Steps 2 through 14.

8.5 Filtering nutrient samples using peristalic method:

1) Unpreserved dissolved nutrients samples should be filtered as close to collection as possible.

2) Put on new latex or nitrile gloves.

3) Assemble and attach pump head to power drive, if not already assembled.

4) Plug power drive into power source.

5) Put on new latex or nitrile gloves.

6) Remove acid-rinsed tubing from plastic bag, taking care to prevent contamination and place in head draping the long end into the churn splitter and dangling the short end out of contact from truck, boat or boat seats.

7) Fill two 500 ml clean sample bottles with DI water.

8) Turn on pump and begin rinsing tubing with a minimum of 250 DI water.

9) As tubing rinses, remove cartridge filter from plastic bag from plastic bag and insert cartridge while pump is still running to the tube's dangling end. Care should be taken to ensure filter cartridge is inserted in correct direction (arrows on side of filter show direction of flow).

10) Rinse 1,000 ml of DI water through the filter cartridge prior to sample filtration. Run DI water through until no more water comes out of the filter.

11) Place the long, draping end of the tubing into the churn splitter ensuring the tubing is adequately submerged in the sample water.

12) Run 250 ml sample water through the filter cartridge.

13) Triple-rinse sample bottle and lid with sample water coming out of the filter cartridge.

14) Fill sample bottle.

15) Preserve the sample with 2 ml 1/5 sulfuric acid or 0.2 ml concentrated sulfuric acid.

16) Place samples in the cooler on ice.

17) If filter cartridge becomes plugged repeat Steps 7 through 14 using an in-line 5.0 µm pre-filter placed in-line prior to 0.45 µm filter.

8.6 Filtering chlorophyll-a and -b samples using a vacuum pump:

1) Homogenize (i.e., churn) the remaining sample in the churn splitter and filter the sample as soon as possible.

2) Triple-rinse the filter apparatus three times with approximately 250 ml of DI water prior to each sample.

3) Load a glass fiber filter in the apparatus and connect the vacuum pump.

4) Using the graduated cylinder, measure out and filter a known volume of sample water. **Note:** Filter enough sample so that the filter is distinctly coated with algae and the flow of water perceptibly slows, a minimum of 1,000 ml is desired but is not always possible.

5) Squirt the sides of the filter apparatus with DI water to wash down any algal or cyanobacterial cells.

6) Allow the filter to dry slightly before removing the top-half of the filter apparatus. Disconnect filter apparatus from filter assembly.

7) Remove the filter from the filter assembly, fold once and place in a 50-ml vial.

8) Wrap the vial in aluminum foil and then place the label on the outside with the volume filtered recorded on the label. Note: Some labs require label to be placed on the vial and then wrapped in foil, so check with the analyzing lab on their preference.

9) Place the wrapped and labeled vial directly on dry ice to avoid degradation of algal cells, if possible. If dry ice is not available, use wet ice and document preservation method.

8.7 Phytoplankton sample collection

1) Homogenize (i.e., churn) the sample.

2) Label sample container and place packing tape over the label.

3) Fill sample container for phytoplankton analysis.

4) Preserve the phytoplankton sample using approximately 2-ml of Lugol's Solution, or until the liquid has the color of "weak tea".

5) Place the sample in the cooler on ice or frozen gel packs.

9.0 DATA AND RECORDS MANAGEMENT

Samplers will fill out the field report form, water column chemistry sample log and Sample ID/Custody Record (all in Appendix A).

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

Quality Assurance and Quality Control (QA/QC) procedures will be followed as explained above. QA/QC samples (i.e., field bottle blanks and duplicates) will be collected at the first and subsequent tenth sample for each project (i.e., 1st, 10th, 20th, etc.). A project-specific Sampling and Analysis Plan (SAP) may require different measures of QA/QC. For example, smaller-scale 319 water quality monitoring projects may not require field bottle blank sample collection.

Related DWQ SOPs

Standard Operating Procedures for the Measurement of Temperature and Dissolved Oxygen Profiles in Lake and Reservoirs

Standard Operating Procedures for Measuring Secchi Disk Transparency

References

NDDEQ. December 2020. Quality Assurance Program Plan for Water Quality and Watershed Projects/Studies. Watershed Management Program, Division of Water Quality, Department of Environmental Quality, State of North Dakota. Bismarck, ND.

APPENDIX A

Field Reporting and Custody Forms

NORTH Dakota | Environmental Quality
Be Legendary.

Mutiple Sample Set
Sheet #_____of_____

Sample Arrival Time-Stamp:_____

CUSTODY RECORD AND ANALYSIS REQUEST – Watershed Management Program

Account #	**Project Code:**	**Project Name:**	**FOR LABORATORY USE ONLY** **Nutrient/Nitrate bottle(s) checked for preservation by:** **Temp of Cooler:**
DEQ Program:	**DEQ Project #:**	**DEQ Cost Center #:**	**Point of Contact/DPM:**
Sampled By:		**Sampler Phone #:**	
Analysis Requested:		***Collection Method: (See Note)**	**Matrix: Soil Water Other (explain)** _____ **Enforcement? Yes No**

Lab ID (Enter # from lids of samples here)	Site ID/STORET #	Sample Location (Lat Long or TRS)	Sample Date	Sample Time	# of Bottles	Cooler #	Co-located Site ID and/or Comments	Depth in meters	Field Measurements	
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH

*** Collection Methods (Record Above):** Depth Integrated (DI) ~ Depth/Width Integrated (DWI) ~ Grab ~ 0-2 meter column
When collecting lake samples, you ***MUST*** include the sampling depth(s).

Relinquished by	Date and Time	Received by	Date and Time

Revised 6/15/20

ED_013266B_00000036-00082

NORTH Dakota | Environmental Quality
Be Legendary.™

Lake Profile Field Log
Division of Water Quality
Watershed Management Program
Phone: 701-328-5210 Fax: 701-328-5200

Project Code:		Project Name:	
Site Identification:		Sampler(s):	
Site Description:			
Date: / /	Time: :	Ambient Temp:	Wind Speed: (mph)
Wind Direction:	% Cloud Cover:	Secchi Disk: (m)	Barometer: (mm/Hg)
Chlorophyll-a:	Phytoplankton:	Initial DO:	Final DO:
Sample Depths: _____ Meters, _____ Meters, _____ Meters, _____ Meters			
Comments:			

Depth (m)	Temp (c)	DO (Mg/L)	pH	Specific Conduct.	Comments

09/22/2020

Checked by: _____ Date: _____

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."

Printed Name	Signature	Date

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature

APPENDIX D
COLLECTION AND PROCESSING OF FISH TISSUE PLUG SAMPLES FOR MERCURY ANALYSIS

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	Joshua Wert	
Program Manager	Aaron Larsen	

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

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 3

4.0 CAUTIONS.....3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES 3

7.0 EQUIPMENT AND SUPPLIES 4

8.0 FIELD PROCEDURE 4

9.0 LABORATORY PROCEDURE.....6

10.0 DATA AND RECORD MANAGEMENT 7

11.0 QUALITY ASSURANCE AND QUALITY CONTROL 7

12.00 REFERENCES.....7

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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.

In general, 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 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 largest and smallest fish within each group should not exceed the average length of the group by more the 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 jell 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 fallowed as explained above. Individual will have to fallow 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

North Dakota Department of Environmental Quality
Division of Water Quality
Fish Collection Field Log
Telephone: 701.328.5210
Fax: 701.328.5200

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

Figure 7.15.1 Fish tissue collection field data form.

North Dakota Department of Environmental Quality
Sample Identification Record
Division of Laboratory Services–Chemistry
Telephone: 701.328.6140
Fax: 701.328.6280

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."

Printed Name	Signature	Date

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature

APPENDIX E
COLLECTION AND PROCESSING OF WHOLE FISH TISSUE SAMPLES

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	Joshua Wert	
Program Manager	Aaron Larsen	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Collection and Processing of Whole Fish Tissue Samples
Type: Standard Operating Procedure 7.13
Version: 3.0
Date: 01/22/2020
Author: Joshua Wert

REVISION HISTORY

Revision	Change Description	Date	Authorization

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 2

4.0 CAUTIONS.....3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES 3

7.0 EQUIPMENT AND SUPPLIES 4

8.0 FIELD PROCEDURE 5

9.0 LABORATORY PROCEDURE.....6

10.0 DATA AND RECORD MANAGEMENT 6

11.0 QUALITY ASSURANCE AND QUALITY CONTROL 7

12.00 REFERENCES.....7

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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.

In general, 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 toxic contamination in our lakes and streams. The data is also used in screening to determine which waterbodies require additional sampling for the possible issuance of fish consumption advisories.

In summary, 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 ± 25 percent of the average length of the entire composite group. The largest fish possible 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
_____ Latex gloves

8.0 FIELD PROCEDURE

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

1. For general survey purposes, 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 ± 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 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 (7.13.2).

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)

10.0 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.

11.0 QUALITY ASSURANCE AND QUALITY CONTROL

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

North Dakota Department of Environmental Quality
Division of Water Quality
Fish Collection Field Log
Telephone: 701.328.5210
Fax: 701.328.5200

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

Figure 7.13.1 Fish tissue collection field data form.

North Dakota Department of Environmental Quality
Sample Identification Record
Division of Laboratory Services–Chemistry
Telephone: 701.328.6140
Fax: 701.328.6280

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 7.13.2 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

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."

Printed Name	Signature	Date

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature

APPENDIX F
COLLECTION AND PRESERVATION OF STREAM AND RIVER GRAB SAMPLES

NORTH Dakota | Environmental Quality
Be Legendary.™

918 E. Divide Avenue
Bismarck, North Dakota 58501
Phone: (701) 328 -5150
Fax: (701) 328-5200

AUTHORIZATIONS

Title	Name	Signature
SOP Author	McKenzie Schick	
Program Manager	Aaron Larsen	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Collection and Preservation of Stream and River Grab Samples
Type: Standard Operation Procedure #7.08
Version: 3.0
Date: 01/13/2020
Author: McKenzie Schick

REVISION HISTORY

Revision	Change Description	Date	Authorization

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

THIS PAGE LEFT INTENTIONALLY BLANK

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY 2

2.0 SUMMARY OF METHOD 2

3.0 HEALTH AND SAFETY WARNING 2

4.0 CAUTIONS.....3

5.0 INTERFERENCES 3

6.0 FIELD PERSONNEL QUALIFICAITONS/RESPONSIBILITIES.....3

7.0 EQUIPMENT AND SUPPLIES 3

8.0 PROCEDURE 4

9.0 DATA AND RECORD MANAGEMENT 8

10.0 QUALITY ASSURANCE AND QUALITY CONTROL 9

11.0 REFERENCES.....10

APPENDIX A. Field Reporting Form

APPENDIX B. SOP Acknowledgement and Training Form

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 preserving stream and river grab samples. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers. Grab samples collected for chemical analysis should be representative of the entire stream or river. To be representative, samples must be carefully collected, properly preserved, and appropriately analyzed. In general, samples should be collected from the main current of the stream or river at 60% of the total stream depth.

2.0 SUMMARY OF METHOD

Grab samples are only collected on low gradient and slow-moving streams. The grab sample can be collected either by wading or by lowering a sampling device such as a Kemmerer sampler, Van Dorn sampler or weighted open bucket from a bridge crossing.

When collecting the sample by wading, enter the stream slightly down current from the appropriate sampling site, then wade to the area with the greatest current. Triple-rinse each sample bottle with stream water prior to collecting the sample. Place lid on sample bottle then submerge to approximately 60 percent of the stream depth, remove the lid and allow the bottle to fill facing towards the current. Replace the lid prior to removing bottle from stream. A small portion of the sample will need to be decanted off prior to preserving and/or placing in cooler. Note: In very shallow streams care must be taken not to contaminate the sample with bottom sediments.

When collecting from a bridge using a Kemmerer or Van Dorn sampler, triple rinse sample device prior to collection, lower the device into the stream and trip the sampler at 60 percent of the total stream depth. In cases where a sample cannot be taken from a bridge, a dipper cup would be used.

3.0 HEALTH AND SAFETY WARNING

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. Samplers should be aware of ice conditions when sampling when sampling during winter months. If ice is dangerous, samples should be taken at a different time.

Field personnel should also be aware of wildlife, insects, and plants that could be harmful as well as heat stroke and hypothermia. A first aid kit should be accessible for any potential cuts, stings, bites, or contact with poisonous plants. Also ensure there is access to water, sunscreen, insect repellant, and extra clothing.

4.0 CAUTIONS

Care should be taken not to disturb sediment and/or substrate during sample collection. Disrupted sediment can give invalid results and plug the filters. (e.g. Geotech inline filters, etc.)

5.0 INTERFERENCES

Note all factors that may affect the water sample such as high winds/wave action, cattle in water, observed flow, water surface, water clarity, water color, water odor, visual algae cover, number of dead fish, present weather, estimated inches of rain fall in past 72 hours, and any other comments that may be of interest.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel collecting and preserving grab 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 personnel 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

- ☐ A non-metallic sampler (e.g., Kemmerer or Van Dorn sampler), with rope marked at 0.5-meter depth intervals and a messenger.
- ☐ Churn splitter
- ☐ Sample containers
- ☐ Acid for sample preservation
- ☐ Sample labels.

- ☐ Clear tape for sample containers
- ☐ Coolers with ice and/or frozen gel pack(s).
- ☐ Deionized water for sample blanks and decontamination.
- ☐ For vacuum method.
    - Vacuum filter holder.
    - Vacuum pump.
    - 0.45 µm membrane filters (Millipore HAWP 047 00 or equivalent).
    - Pre-filters (Millipore AP40 0047 05 or equivalent).
    - Stainless steel forceps.
- ☐ For peristaltic method.
    - Power Drive (Compact Cat No. P-07533-50 or equivalent)
    - Peristaltic head (Easy Load II Cat No. P-77200-62 or equivalent).
    - In-line 0.45 µm cartridge filters (Geotech dispos-a-filter or equivalent).
    - In-line 0.70 µm cartridge pre-filters (Geotech dispos-a-filter or equivalent).
    - Tubing (Masterflex silicone Cat No. P-96400-24 or equivalent).
- ☐ Field report form.
- ☐ Sample ID/Custody Record
- ☐ Black ballpoint pen or pencil
- ☐ Sample and blank log forms
- ☐ Power ice auger (winter sampling)
- ☐ Ice skimmer (winter sampling)
- ☐ Sled (winter sampling)

8.0 PROCEDURE

Stream Sample Collection Wading

1. Place a label on each sample container and use clear tape to secure the label to the container. Note: Add information to the cap (e.g., number of sample; analysis type) to make the sample identifiable if the label were to fall off)

2. Triple rinse each sample bottle using stream water. Note: Do not rinse the fecal coliform bacteria or the pesticide sample bottles.

3. Fill the sample bottle: Samples should be collected in the main current at that depth which is approximately 60 percent of the total water depth. Wade to the stream sampling location and inserting sample container facing against the current, allowing it to fill naturally at the appropriate depth. At greater water depths, an appropriate sampling device should be used. Note: Care should be taken so that the sample is not contaminated by disturbing the stream bed upstream from the collection point.

4. Preserve the sample containers appropriately with sulfuric or nitric acid and place samples in a cooler on ice.

5. Fill out the Sample ID/Custody Report (Appendix A) and the water chemistry sample log (Appendix A).

Stream Sample Collection using Dipper Cup

1. Place a label on each sample container and use clear tape to secure the label to the container. Note: Add information to the cap (e.g., number of sample; analysis type)to make the sample identifiable if label were to fall off.

2. Triple rinse dipper cup and churn splitter using stream water.

3. Fill the churn splitter using the dip cup: Samples should be collected in the main current. Note: Care should be taken so that the sample is not contaminated by disturbing the stream bed upstream from the collection point.

4. Sample bottles will be triple rinsed and filled from the churn splitter. Any samples

that need to be filtered will be filtered from the same churn splitter.

5. Preserve the sample containers appropriately and place samples in a cooler on ice.

6. Fill out the Sample ID/Custody Report (Appendix A) and the water chemistry sample log (Appendix A).

Stream Sample Collection using Van Dorn

1. Place a label on each sample container and use clear tape to secure the label to the container. Note: Add information to the cap (e.g., number of sample; analysis type)to make the sample identifiable if label were to fall off)

2. Triple rinse Van Dorn and churn splitter using stream water.

3. Fill the churn splitter using the Van Dorn: Samples should be collected in the main current on the bridge. Samples should be collected in the main current at 60 percent of the total water depth. Note: Care should be taken so that the sample is not contaminated by disturbing the stream bed upstream from the collection point.

4. Sample bottles will be triple rinsed and filled from the churn splitter. Any samples that need to be filtered will be filtered from the same churn splitter.

5. Preserve the sample containers appropriately and place samples in a cooler on ice.

6. Fill out the Sample ID/Custody Report (Appendix A) and the water chemistry sample log (Appendix A).

Stream Sample Filtration Vacuum Method

1. Total dissolved phosphorus samples should be filtered immediately.

2. Put on new latex surgical gloves.

3. Remove filter holder from the plastic bag and assemble.

4. Rinse filter apparatus with stream water.

5. Rinse the filter with 1,000 mL of DI water.

❖ <u>If Pre-filter is needed</u>

- Load a pre-filter in the filter apparatus and connect the vacuum pump.
- Leach the filter with 1,000 ml of DI water.
- Filter the sample through the pre-filter. Place the sample back into the sample container.
- Load a 0.45 µm filter into the filter apparatus and connect the vacuum pump.

5. Filter the sample through the 0.45 µm filter.

6. Triple rinse the sample container with deionized water.

7. Transfer the filtered sample back into the sample container.

8. Preserve the sample with 2 mL 1/5 sulfuric acid or 0.2 mL concentrated sulfuric acid lowering the pH to 2 or less.

9. Place the preserved sample in the cooler on ice.

10. If additional samples require filtration, repeat Steps (3) through (9).

<u>Field Sample Filtration Peristaltic Method</u>

1. Assemble and attach pump head to power drive.

2. Plug in power drive.

3. Put on new latex surgical gloves.

4. Remove acid rinsed tubing from plastic bag, taking care to prevent contamination and place in head draping a long end into the churn splitter and dangling the short end out of contact with anything.

5. Turn on pump and begin rinsing tubing with a minimum of 1,000 mL of DI water.

6. As tubing rinses remove cartridge filter from plastic bag and insert cartridge to the tube's dangling end while pump is still running. Care should be taken to ensure filter cartridge is inserted in the correct direction, by noting directional arrow on side of filter.

7. Run 250 ml of sample water through cartridge filter.

8. Place labels on bottles.

9. Triple rinse the sample bottles and lids with sample water coming out of the filter cartridge.

10. Fill sample bottles.

11. Preserve nutrient sample with 2 ml 1/5 sulfuric acid or 0.2 ml concentrated sulfuric acid and ICP Metals or Trace metals with 2 ml concentrated nitric acid lowering the pH to 2 or less. Note: Dissolved minerals are not preserved.

12. Place samples in the cooler on ice.

13. Replace hose in bag to avoid contamination.

If cartridge becomes plugged repeat Steps (6) through (13) with an in-line 2.0 µm pre-filter placed in-line prior to the 0.45 µm filter.

9.0 DATA AND RECORDS MANAGEMENT

Data collected will be recorded on the field form (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 reading such as high winds/wave action, cattle in water, observed flow, water surface, water clarity, water color, water odor, visual algae cover, number of dead fish, present weather, estimated inches of rain fall in past 72 hours, and any comments. Field forms and notes should be stored in the appropriate project folder at DWQ.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

Stream Blank Sample Collection

1. Field blank samples are collected with the first and every tenth stream sample collected
   (i.e., 1, 10, 20....). If the sample log indicates a blank sample should be collected, follow the steps below.

2. Place a label on each sample container and fill out the sample information log form (Appendix A). Note: Field sample blanks should be identified with STORET number 389990. Be sure to indicate on the label the project name and type of sample being duplicated.

3. Using DI water, triple-rinse each sample bottle.

4. Fill each bottle with DI water. Note: No blank is done for E. coli.

5. Samples that need to be filtered will be done identically, including rinsing the filter with 1000 mL of DI water.

5. Preserve each sample appropriately. Note: Do not preserve the total dissolved phosphorus sample.

6. Place the sample in a cooler on ice.

Stream Duplicate Sample Collection

1. Similar to blanks, duplicate samples are collected with the first and every following tenth stream sample collected (i.e., 1st, 10th, 20th....). If the sample log indicates a duplicate sample should be collected, follow the steps below.

2. Place a label on each sample container and fill out the Sample ID/Custody Report (Appendix A). Note: Duplicate samples should be identified with STORET number 389999. Be sure to indicate on the label the project name and type of sample being duplicated.

3. Collect the sample following steps in the procedure for Stream Sample Collection.

4. Place the samples in a cooler on ice.

11.0 REFERENCES

INTERAGENCY FIELD MANUAL FOR THE COLLECTION OF WATER-QUALITY DATA Collecting Water-Quality Samples. USGS https://pubs.usgs.gov/of/2000/ofr00-213/manual_eng/collect.html.

APPENDIX A

Field Reporting Form

CUSTODY RECORD AND ANALYSIS REQUEST – Watershed Management Program

Account #	Project Code:	Project Name:		FOR LABORATORY USE ONLY Nutrient/Nitrate bottle(s) checked for preservation by: Temp of Cooler:
DEQ Program:	DEQ Project #:	DEQ Cost Center #:	Point of Contact/DPM:	
Sampled By:		Sampler Phone #:		
Analysis Requested:		*Collection Method: (See Note)	Matrix: Soil Water Other (explain) _____	Enforcement? Yes No

Lab ID (Enter # from lids of samples here)	Site ID/STORET #	Sample Location (Lat Long or TRS)	Sample Date	Sample Time	# of Bottles	Cooler #	Co-located Site ID and/or Comments	Depth in meters	Field Measurements	
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH
									Temp °C	DO mg/L
									SC µ	pH

*** Collection Methods (Record Above):** Depth Integrated (DI) ~ Depth/Width Integrated (DWI) ~ Grab ~ 0-2 meter column
When collecting lake samples, you ***MUST*** include the sampling depth(s).

Relinquished by	Date and Time	Received by	Date and Time

River and Stream Sampling Field Log
North Dakota Department of Environmental Quality
Division of Water Quality - Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date: ___/___/____	Temperature	DO	
		Time: :	SC	pH	

Water Quality Sampling Field Log
North Dakota Department of Environmental Quality
Division of Water Quality – Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Sample Number	Station ID and Description	Depth	Date	Time	QA/QC		Initials	Comments
					DUP	BLK		

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."

Printed Name	Signature	Date

SOP Acknowledgement and Training Form (cont.)

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.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature