



Environmental Quality

4201 Normandy St.
Bismarck, North Dakota 58503
Phone: (701) 328 -5150
Fax: (701) 328-5200


AUTHORIZATIONS

Title	Name	Signature
SOP Author	Joe Nett	N/A
Program Manager	Joshua Wert	

QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

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1.0 SCOPE AND APPLICABILITY

This document presents the North Dakota Department of Environmental Quality, Division of Water Quality's (DWQ) Standard Operating Procedure (SOP) for collecting and 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 peristaltic method
 - _____ Power Drive (Compact Cat no. P-07533-50 or equivalent)
 - _____ Peristaltic 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 (10% potassium iodide, 5% iodine)

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 (Appendix A), Sample ID/Custody Record and the water column chemistry sample log (Appendix A).
- 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 peristaltic 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

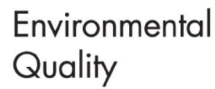
NDDEQ. December 2020. Quality Assurance Program Plan for Water Quality and Watershed Projects/Studies. Watershed Management Program, Division of Water Quality, Department of Environmental Quality, State of North Dakota. Bismarck, ND.

APPENDIX A
Field Reporting and Custody Forms

Sample Arrival Time-Stamp: _____

CUSTODY RECORD AND ANALYSIS REQUEST – Watershed Management Program

Account #		Project Code:		Project Name:		<div style="border: 2px solid black; padding: 5px;"> FOR LABORATORY USE ONLY Nutrient/Nitrate bottle(s) checked for preservation by: _____ Temp of Cooler: _____ </div>		Enforcement? Yes No	
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)					
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 pH
									Temp °C DO mg/L SC pH
									Temp °C DO mg/L SC pH
									Temp °C DO mg/L SC pH
									Temp °C DO mg/L SC pH
									Temp °C DO mg/L SC pH
									Temp °C DO mg/L SC pH
<p>* 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).</p>									
Relinquished by			Date and Time			Received by			



Division of Water Quality

Phone: 701-328-5210 Fax: 701-328-5200

Checked by: _____ Date: _____

09/22/2020

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:	Collecting and Preserving Lake Water Quality Samples
Document Version Number:	1.1
Document Version Date:	02/08/2024

Please sign below in accordance with the following statement:

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

[illegible]

SOP Acknowledgement and Training Form (con't)

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

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

[illegible]