

Standard Operating Procedure #7: Samples and Analytical Laboratory Requirements

Version 1.3 (2021)

In Water Quality Monitoring Protocol for Inland Lakes: Great Lakes Inventory and Monitoring Network

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Revision History Log

The following table lists all edits and amendments to this document since the original publication date. Information entered in the log must be complete and concise. Users of this standard operating procedure will promptly notify the project manager and/or the Great Lakes Network (GLKN) data manager about recommended and required changes. The project manager must review and incorporate all changes, complete the revision history log, and change the date and version number on the title page and in the header of the document file. For complete instructions, please refer to Revising the Protocol, SOP #13.

Previous Version #	Revision Date	Author (with title and affiliation)	Location in Document and Concise Description of Revision	Reason for Change	New Version #
1.0	2/25/2010	Rick Damstra, David VanderMeulen, Joan Elias, GLKN	7.2 Sample Handling and Processing – clarification; 7.3 Shipping Procedures – clarification; 7.4 Quality Assurance/Quality Control - clarification	To bring SOP into alignment with procedures practiced	1.1
1.1	5/01/2015	David VanderMeulen, GLKN	Updated the NPS contact author, added that field samples might be collected as elbow deep grab samples, included information about acid-washing bottles, fixed a handful of grammatical errors throughout	To clarify and update SOP based on changes that have occurred since Version 1.1 was published in 2010	1.2
1.2	3/2021	Alexander Egan, Aquatic Ecologist, GLKN	Minor text changes.	To clarify and update SOP based on changes that have occurred since Version 1.2 was published in 2015	1.3
Add rows as needed for each change or set of changes tied to an updated version number					

Acknowledgements

Combinations of existing protocols were used to develop this standard operating procedure for handling and processing water samples prior to sending them to an analytical laboratory. The authors wish to acknowledge these sources (Hoffman et al. 2005, O’Ney 2005, USGS 2005, WOW 2004, Penoyer 2003, EPA 2001, and Baker et al. 1997) for the guidance they provided. Thanks also go to White Water Associates for providing examples of forms for analytical requests and chain of custody.

7.0 Introduction

As part of our water quality monitoring program, we will collect water samples for analysis of basic limnological parameters that may respond to stressors such as atmospheric deposition, land use change, and recreational pressures. Because neither the GLKN nor the individual park units currently have certified laboratories and instrumentation for performing these analyses, water samples will need to be processed soon after collection for transport to a suitable laboratory. This will require filtering and preserving subsamples of water and filters according to methods specific to the analyte. This standard operating procedure (SOP) is designed to provide detailed instructions on the handling and processing of water samples prior to analysis by an analytical laboratory.

We will collect water in the field (SOP #6) for the analysis of the following parameters:

- Nutrients: total phosphorus (TP), total nitrogen (TN), nitrate+nitrite-N ($\text{NO}_3+\text{NO}_2\text{-N}$), and ammonium-N ($\text{NH}_4\text{-N}$)
- Total chlorophyll-*a*
- Major anions (SO_4^{-2} and Cl^-);
- Alkalinity
- Major cations (Mg^{+2} , Ca^{+2} , Na^+ , K^+)
- Dissolved silica (SiO_2)
- Dissolved organic carbon (DOC)

Water chemistry will be performed by one or more analytical laboratories that have demonstrated the ability to measure analytes at detection levels adequate to meet our needs. Preferably, the laboratories will be state- or federally-certified for performing the above water chemistry analyses in natural waters, or an academic research laboratory that can demonstrate quality assurance and quality control (QA/QC) procedures consistent with SOP #12 and current EPA procedures used as the basis for state certification of commercial environmental laboratories. The GLKN's preference is for laboratories to provide clean sample bottles and preservatives, where appropriate, as well as chain of custody documentation and sample logging.

7.1 Summary of Analytical Methods

Analytical methods, method detection limits, and procedures related to handling samples change over time as the science progresses. Table 1 summarizes examples of the variety of methods, detection limits, preservation techniques, and holding times for water samples addressed in this protocol. Table 2 summarizes the frequency at which water samples will be collected for analysis.

Table 1. Examples of analytical methods, method detection limits (MDLs), containers, and holding times.

Analyte	Analytical Note 1	Method #	Det. Limit	Vol. (mL)	Filter	Preservation	Sample Bottle Note 2	Hold Time
Alkalinity	Titrimetry	310.1 EPA-NERL	10 mg/L			4°C		14 d
	Spec. auto.	310.2 EPA-NERL	10 mg/L			4°C		14 d
	Titrimetry	NFM USGS-OWQ	0.01 meg/L		Note 4	None		none
	ICP	3120B APHA	10 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
Calcium	Titrimetry	215.2 EPA-NERL	0.5 mg/L		Note 3	4°C		6 mo
	FAA	I-3152 USGS-NWQL	0.1 mg/L	250 mL	Note 3	pH<2 HNO ₃	P	180 d
Chloride	IC	300.0 EPA-NERL	0.02 mg/L			4°C	P or G	28 d
	Colorimetry	325.2 EPA-NERL	1 mg/L			4°C		28 d
	Titrimetry	4500-Cl APHA	0.15 mg/L	100 mL		4°C	P or G	28 d
Chlorophyll-a	Spect.	10200 APHA	2 ug/L	≤1 L	Note 4	Freeze filter	P	30 d
Color	Spect.	110.2 EPA-NERL	5 Pt units		Note 5	4°C	G	48 hr
	Vis. Comp.	I-1250 USGS-NWQL	1 Pt-co	250 mL	Note 5	4 °C	P	30 d
DOC	Spect.	415.3 EPA	0.018 mg/L	125	Note 3	pH<4 H ₂ SO ₄	G	28 d
	Spect.	0-1122-92 USGS	0.1 mg/L			4°C	AG	
K	ICP	3120B APHA	0.3 mg/L		Note 3	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	5 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
Mg	ICP	3120B APHA	20 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	0.5 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
Na	ICP	3120B APHA	30 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	2 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
NH ₄ -N	Selective elec.	4500-NH ₃ E	0.08 mg/L			4°C/pH2,0°C		24 hr / 28 d
	Colorimetry	350.2 EPA-NERL	0.08 mg/L			pH<4 H ₂ SO ₄		28 d
	Titrimetry	4500-NH ₃ APHA	5 mg/L			4°C / pH2,0°C		24 hr / 28 d
SiO ₂	ICP	3120B APHA	20 ug/L		Note 3	pH<2 HNO ₃	P or G	6 mo
	Spect.	4500- SiO ₂ D APHA	0.04 mg/L		Note 3	No, 4°C	P	28 d
	FIA-Spect.	4500- SiO ₂ F APHA	0.78 ug/L		Note 3	No, 4°C	P	28 d
TSS	Gravimetric	I-3765 USGS-NWQL	1 mg/L	250–500	Note 4	4°C filter	P	NA

Analyte	Analytical Note 1	Method #	Det. Limit	Vol. (ml)	Filter	Preservation	Sample Bottle Note 2	Hold Time
SO ₄	IC	4110C APHA	75 ug/L		Note 3	pH<4 H ₂ SO ₄	P or G	
SO ₄ (continued)	CIE-UV	D6508 ASTM	0.1 mg/L		Note 3	pH<4 H ₂ SO ₄		ASAP
	Spect.	37512 EPA-NERL	0.5 mg/L		Note 3	pH<4 H ₂ SO ₄	P or G	28 d
TP	Spect.	I-2606 USGS-NWQL	0.001 mg/L	125 mL		MgCl 4°C	BrownP	30 d
	Alkaline P	USGS 2003	0.01 mg/L	120 ml	Note 6	4°C / H ₂ SO ₄		48 hr / 30 d
	ICP	200.7 EPA-NERL	60 ug/L			pH<2 HNO ₃	P	6 mo
TN	Alkaline P	USGS 2003	0.03 mg/L	120 ml	Note 6	4°C / H ₂ SO ₄		48 hr / 30 d
	Titrimetry	4500-N	0–100 mg/L			4°C	AG	7 d
	Combustion	440.0 EPA-NERL	0.1 mg/L			Filter		100 d

Source: National Environmental Methods Inventory website (NEMI 2006)

This list is not an endorsement of any particular method or laboratory for any particular analyte. Rather it is to be used as a reference for the range of analytical methods available for each analyte. There are surface water conditions (pH, turbidity, other elements) that make a particular method unsuitable for a particular situation. As GLKN is monitoring surface water, the methods listed were chosen as representative of the lower range of detection limits.

Note 1. CIE-UV= capillary ion electrophoresis with UV detection, FAA = flame atomic absorption, FIA = flow injection analysis, IC= ion chromatography, ICP = inductively coupled plasma, Spec. auto = spectroscopy with autoanalyzer

Note 2. P = plastic (polypropylene), G=glass, AG=amber glass

Note 3. 0.45µm membrane filter. Pre-filter for dissolved portion analysis.

Note 4. 0.45µm glass fiber filter.

Note 5. 0.45µm membrane filter or centrifuge is recommended to remove suspended solids that affect color; however some color will also be removed.

Note 6. USGS 2003. Evaluation of Alkaline Persulfate Digestion as an Alternative to Kjeldahl Digestion for Determination of Total and Dissolved Nitrogen and Phosphorus in Water By Charles J. Patton and Jennifer R. Kryskalla. U.S. Geological Survey Water-Resources Investigations Report 03-4174.

Table 2. Frequency of analyses of water samples and depths at which samples will be collected. Surface samples will be a 0–2 m integrated sample or an elbow-deep (ca. 0.3 m) grab sample. Bottom samples will be collected during mid-summer if a lake is stratified.

Water Quality Variables	Monitoring Frequency
Total phosphorus (TP)	3x/yr, surface 1x/yr, bottom
Total Nitrogen (TN)	3x/yr, surface
Nitrate + Nitrite-Nitrogen (NO ₃ +NO ₂ -N)	3x/yr, surface
Ammonium-Nitrogen (NH ₄ -N)	3x/yr, surface

Water Quality Variables	Monitoring Frequency
Major Anions (SO_4^{2-} , Cl^-)	1x/yr; surface
Alkalinity	1x/yr; surface
Major Cations (Mg^{+2} , Ca^{+2} , Na^+ , K^+)	1x/yr; surface
Dissolved silica (SiO_2)	1-2x/yr; surface
Dissolved organic carbon (DOC)	3x/yr; surface
Total chlorophyll-a	3x/yr; surface

7.2 Sample Handling and Processing Procedures

The following general techniques will be observed throughout the procedures detailed in 7.2.1 through 7.2.4.

1. Keep all water samples cool and dark until processing is complete and samples are shipped to the analytical laboratory.
2. Use only new, clean sample bottles supplied by the analytical laboratory, purchased pre-cleaned from a supplier, or those cleaned and acid-washed with 0.1N HCl by GLKN staff.
3. Rinse filtration equipment with deionized water (DIW) three times between samples.
4. Avoid touching the inside of sample bottles and filtering apparatus, tips of forceps, and filters to prevent contamination of the samples.
5. When filtering samples in the field, use an enclosed filtering apparatus to minimize contamination from airborne sources.
6. Wear disposable, powderless gloves when working with acids and other preservatives.
7. Filter samples in the order of anticipated phosphorus concentrations, from low to high. After filtering a water sample that is expected to contain high nutrient concentrations, rinse the apparatus three times with 0.1N HCl followed by three times with DIW water before processing the next sample.
8. Prepare QA/QC samples in the same manner as regular samples, using water from the same sample collection container.
9. Rinse all reusable equipment with DIW immediately, before equipment dries.
10. Ensure all sample bottles are labeled correctly, completely, and legibly.

11. Check laboratory equipment and supplies list (Table 3) and ensure equipment is clean and ready for use and supplies are adequate.
12. Prepare a temperature check bottle for each anticipated cooler, if recommended by the contract analytical laboratory. Use tap water to fill an extra bottle of the same size used for one of the analytes and label as “Temperature Check.” Store this check bottle in refrigerator with other samples; package and send to the analytical laboratory with the other samples.

Table 3. Laboratory equipment and supplies list.

• Filtration towers and manifold (4.7 mm) plastic • Vacuum pump with pressure gauge and extra filtering flask as a water trap to protect the pump in case of overflow • Graduated cylinders, plastic 250, 500 and 1000 mL • Whatman GF/C filters (4.7 cm diameter) • 0.45 µm Millipore membrane filters (4.7 cm diameter) • Filter forceps with broad tips • Aluminum foil • Labeling tape, permanent markers • Deionized water (ASTM grade 1 or 2; 1–10 megohm) • Acid for preservation (according to contract laboratory specifications) • Freezer • Plastic storage bags • Sample bottles (provided by analytical laboratory) • Insulated ice chest, ice, and ice packs • Saturated MgCO₃ solution (for chlorophyll-a), depending on laboratory method • Adjustable automatic pipettes: 1–5 mL; 0.2–1 mL; 0.02–0.1 mL • Paraffin paper roll • Wash (squirt) bottles – 500 mL • Kim wipes
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The following sections detail the procedures to be followed when processing water samples for particular analysis.

7.2.1 Total Chlorophyll-a

1. Fit rinsed filtering device with a Whatman GF/C glass fiber filter using forceps, smooth side down (curl is up).
2. Agitate water sample (always shake well to minimize subsampling error for solids).
3. Set pump vacuum to ≤0.5 atmospheres (7.5 PSI or 380 mm Hg). If using a hand pump, maintain pressure at or below 10 PSI.
4. Use a glass or plastic graduated cylinder to measure 100–1000 mL of water sample. Filter sample. If water is very turbid, filter small aliquots (100 mL) to avoid clogging the filter.

Sufficient volume has been filtered when a green, brown, or tan color is clearly visible on the filter and the flow decreases to a few drops/second.

5. Add 0.15 mL (approx. 3 drops) of saturated MgCO_3 during the last 30 mL of filtering to buffer the filter, if required by the method used by the contract analytical laboratory.
6. Rinse graduated cylinder and filtering apparatus with DIW and pass through filter to include any algae that may have adhered to the sides of the cylinder.
7. Record volume filtered on data sheet (excluding DIW rinse).
8. Use forceps to fold filter into quarters with sample on the inside; do not touch filter with fingers.
9. Wrap filter in foil; label foil with sample location, date, and time sample was collected, and volume filtered. Place foil in small, sealable baggie with standard laboratory label.
10. Refrigerate immediately and freeze as soon as possible. Place small baggies with foils together in a large, sealable freezer bag. A third watertight container may be used for shipping to ensure that melt water in transport will not corrupt the samples.

7.2.2 Unfiltered (Raw) Samples

1. Rinse sample bottle provided by analytical laboratory 1x with sample water.
2. Fill sample bottle with sample water (fill to neck if sample will be frozen).
3. Refrigerate or freeze, as per laboratory instructions, until packaging for transport to analytical laboratory.

7.2.3 Filtered Samples

1. Using clean forceps, place a $0.45\mu\text{m}$ Millipore cellulose membrane filter in the filtration apparatus. Rinse with 100 mL DIW into a cleaned (0.1N HCl and DIW rinsed as per sample bottle cleaning) filtering flask (glass or plastic). Rinse flask with filtrate and discard filtrate.
2. Filter a small amount (approx. 50 mL) of sample water; rinse filtering flask with filtrate and discard filtrate.
3. Filter enough of the sample to produce the required amount of filtrate to be tested.
4. Dispense the filtrate into separate bottles provided by the analytical laboratory as follows:
 - Unpreserved constituents—rinse bottle with small amount of filtrate (approx. 10 mL) and discard; fill bottle to neck, refrigerate or freeze as per laboratory instructions.
 - Preserved constituents—if pre-loaded with preservative by the analytical laboratory, fill bottle, and store at room temperature or refrigerate. If bottle does not come pre-loaded with preservative, rinse bottle with small amount of filtrate (approx. 10 mL) and

discard, fill bottle approximately $\frac{3}{4}$ full, add the preservative (ampule provided by laboratory) and continue to fill bottle until full. Gently roll bottle to mix. Store at room temperature or refrigerate.

7.3 Shipping Procedures

1. Call FedEx or other courier service ahead of time to arrange pick-up.
2. Make large quantities of ice cubes and ice blocks (or buy ice) ahead of time.
3. Line cooler with large plastic garbage bag.
4. Place all total chlorophyll-*a* baggies containing aluminum foil wrappers in one large sealable plastic bag. Place this baggie between 2 ice packs or bags of ice. It is critical that melt water does not soak the filters, so you may want to place large sealed bag of foil wrappers in a sealed plastic jar before surrounding with ice.
5. Use ice cubes, doubly bagged in plastic bags, to pack around samples; use other ice blocks (water bottles, soda bottles, etc.) as they will fit.
6. Include a temperature check bottle with the sample bottles in each cooler, if used by the contract analytical laboratory.
7. Complete the chain of custody (COC) form, keeping the “client copy” for the project files. Seal the laboratory’s copies in a one-gallon plastic sealable bag and tape to the inside cover of the cooler. Prepare separate COC forms for each cooler. An example COC form is provided in Attachment B.
8. If the refrigerated samples are sent in the same insulated cooler with the frozen samples, protect them from freezing by wrapping them in newspaper, bubble wrap, or something similar.
9. Ship samples overnight so they are received the following day during a work-week, whenever possible. Contact the laboratory about Saturday shipment receipt availability before shipping samples on a Friday. Many laboratories do not have sample receipt staff on Saturday or charge extra for staff time.
10. Alert the contract laboratory when samples have been shipped via phone or e-mail. Be sure to get acknowledgement from the lab that they know the samples are en route. Ask the lab to let you know when the samples are received.

7.4 Quality Assurance/Quality Control

Quality assurance/quality control refers to all those things good investigators do to ensure their measurements are accurate (the absolute true value) and precise (reproducible, consistent), and that they include a good estimate of their uncertainty. It specifically involves following established rules in the field and laboratory to assure everyone that the sample is representative of the site, free from outside contamination, and that it has been analyzed following standard QA/QC methods. These methods typically involve comparing the sample to a set of known samples for estimating accuracy and replicating the measurement to estimate its precision.

In the context of field sampling and processing, quality assurance protocols are meant to ensure that data collected are as representative of the lake or stream site as possible. Such procedures include implementing good field procedures and quality-control checks, careful post-collection processing of water samples to minimize artifacts due to contamination or mislabeling, proper storage and preservation techniques while in transit to a laboratory, and proper QA/QC by the laboratory itself. See SOP #12, Quality Assurance/Quality Control, for more details.

Quality assurance at the batch level (within the laboratory) is accomplished by using proper techniques and replicating 10% of the field samples.

The most important aspects of quality control in the collection of water quality samples are: 1) samples collected should represent the lake site at the time the samples are collected, such that the samples produce the quality of information necessary to meet the objectives of the survey; and 2) the integrity of the samples collected is not compromised by contamination, misidentification, or improper sample handling or preservation.

To help meet these quality control aspects, the transport and tracking of the samples from the field to the analytical laboratory that performs the chemistry analyses is critical. Each set of samples should include a Chain of Custody Form (COC), and if the lab uses it, an Analytical Services Request (ASR) (see Appendix B for an example). To ensure correct processing of samples, the information recorded on the COC and ASR forms must correspond to each sample in the shipment. Check with individual analytical laboratory requirements for correct labeling codes and procedures. To prevent water damage to paperwork accompanying samples to the laboratory, place all paperwork inside a sealable plastic bag and tape the bag to the underside of the cooler lid. Keep a copy of the completed COC and ASR forms in the office binder.

7.5 Data and Records Management

Complete and accurate record keeping of field-derived data is an essential component of monitoring water quality. Field technicians, crew leaders, and project leaders share responsibility for collecting, verifying, and documenting data according to the guidelines in this monitoring protocol and all applicable standard operating procedures. Data and records management include the following responsibilities:

1. Refer to the GLKN Data Management Plan (Hart and Gafvert 2006) for overall guidance.
2. Follow the QA/QC procedures in SOP #8 for specific instructions on data entry, management, verification, and validation.
3. Record and verify observed or measured data values, including completing paper forms and entering data into NPSTORET and/or other electronic databases. NPSTORET maintains the necessary relationships between data values, equipment configuration and calibration, procedures, methods, and metadata.
4. Schedule and perform regular data transfer and backup. Data will be protected from loss or damage by daily backup when possible, or on a feasible schedule approved by the project leader and the data manager.
5. Review, verify, and correct field data and sample processing information as soon as possible after the actual survey (see SOP #8 for more details).
6. Prepare data and procedural documentation, especially deviations from the protocol or study plan, including metadata forms in NPSTORET and additional documentation requested by the project manager or data manager.
7. Ensure that field forms, field notebooks, and other hardcopy records are secure, organized, and available for viewing, reproduction, or transfer upon request.

7.6 Literature Cited

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