

Standard Operating Procedure #12: Quality Assurance/Quality Control

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	David VanderMeulen, Joan Elias, Rick Damstra, GLKN	Table 4 – update detection levels required; info on optical DO probes added; Table 9 – clarify & update calibration frequency	To bring SOP into alignment with procedures practiced	1.1
1.1	5/01/2015	David VanderMeulen, GLKN	Updated the NPS contact author, added language about the use of web-based visualization and data summary tools, continuous monitoring with thermal arrays, data certification, and Aquarius database, updated the number of index lakes in Table 1, added Attachment C, Sample Data Certification Form, 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; added equation numbering.	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					

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12.0 Purpose

This standard operating procedure (SOP) defines procedures for quality assurance and quality control to be used with the Great Lakes Network protocol for monitoring water quality in inland lakes. Quality assurance is the planned and systematic pattern of all actions necessary to provide adequate confidence, or assurance, that a project outcome optimally fulfills expectations. Quality control is the systematic evaluation of the various aspects of a project to ensure, or control, that the standards of quality are being met. Quality control involves specific tasks undertaken to determine the reliability of field and laboratory data. Together, quality assurance/quality control (QA/QC) is a substantial part of any monitoring program. The objective of QA/QC is to ensure that the data generated by a project are meaningful, representative, complete, precise, comparable, scientifically defensible, and reasonably free from bias (Irwin 2006).

12.1 Applicability

Procedures in this SOP will be implemented during all work pertaining to monitoring water quality in inland lakes as described in the protocol narrative. This SOP is designed to assure that all data obtained will contribute quality information to an understanding of the ecological integrity of park units of the Great Lakes Network.

The rationale for dividing a sampling protocol into a protocol narrative with supporting SOPs is based on the following:

- The protocol narrative is a general overview of the protocol that gives the history and justification for doing the work and an overview of the sampling methods, but does not provide all methodological details. The protocol narrative will only be revised if major changes are made, such as changes in sampling design.
- The SOPs are specific step-by-step instructions for performing a given task. They are expected to be revised more frequently than the protocol narrative.
- Usually, when a SOP is revised, it is not necessary to revise the protocol narrative to reflect the specific changes made to the SOP. All versions of the protocol narrative and SOPs will be archived.

The steps for changing the protocol (either the protocol narrative or the SOPs) are outlined in Procedures for Revising the Protocol (SOP #13).

12.2 Summary

Inland lakes are important and valuable resources at six of the parks of the Great Lakes Network (APIS, INDU, ISRO, PIRO, SLBE and VOYA). Lakes at these parks are used extensively by visitors for fishing, boating, swimming, and other recreational activities. The preservation of lake water quality and quantity is of utmost importance to park managers, researchers, and the general public. Monitoring basic water quality ranked among the highest of the network's vital signs (Route 2004).

A national review panel assembled by the NPS Water Resources Division (WRD) recommended a suite of five parameters be measured across all NPS monitoring networks (NPS 2002). In addition to these five mandated parameters (temperature, pH, specific conductance, dissolved oxygen, and flow/water level) we added a measure of clarity (secchi depth or transparency depth) to our core suite. The core suite was ranked highest among potential vital signs for aquatic systems of GLKN parks, although it was recognized that these measurements were less diagnostic of water quality degradation than biotic communities and other water quality variables, such as nutrient concentrations.

Inputs of excess nutrients, invasion and spread of exotic species, and contaminants from atmospheric fallout and surface runoff, and how these stressors affect the chemical and biological functions of lakes are key issues of concern. By monitoring an advanced suite of parameters (nitrogen and phosphorus species, dissolved organic carbon, major ions, alkalinity, dissolved silica, and chlorophyll-*a*), we will provide data for a thorough understanding of changes in lakes over time.

Our overall goal is to develop a program for monitoring water quality in inland lakes that will contribute to an understanding of the health of ecosystems in park units of the Great Lakes Network. The monitoring protocol is intended to document water quality status and trends for individual lakes and provide an indication of status and trends on a park-wide and network-wide basis. The protocol includes analysis of historical data, sample design, field and laboratory methods, data analysis and reporting, and training and operational requirements.

12.3 Definitions and Abbreviations

APIS	Apostle Islands National Lakeshore (Wisconsin)
INDU	Indiana Dunes National Park (Indiana)
ISRO	Isle Royale National Park (Michigan)
PIRO	Pictured Rocks National Lakeshore (Michigan)
SLBE	Sleeping Bear Dunes National Lakeshore (Michigan)
VOYA	Voyageurs National Park (Minnesota)
Blanks	Analytical quality control samples analyzed in the same manner as site samples.
Equipment blank	<i>Also known as field blank.</i> A sample of distilled, deionized water taken to the field, opened, and used as sample water would be (i.e., poured through equipment used to handle samples).

Trip blank	Used to indicate potential contamination due to migration of volatile organic chemicals (VOCs) from the air on the site or shipment into the sample vial. Consists of laboratory distilled, deionized water in a 40 mL glass vial sealed with a Teflon septum and is unopened in the field.
Laboratory calibration blank	Distilled, deionized water injected directly into an instrument; indicates contamination in instrument or source of water.
Laboratory reagent blank	<i>Also known as</i> method blank. Distilled, deionized water manipulated as if it were a sample (digestions, extractions, etc.).
CCV	Continuing Calibration Verification
EPA	Environmental Protection Agency
EMAP	Environmental Monitoring and Assessment Program
GLKN	Great Lakes Inventory and Monitoring Network
m	Meter
MDL	Method Detection Limit
mg/L	Milligrams per liter
ML	Method Limit of quantitation
MQO	Measurement Quality Objective
NAWQA	National Water Quality Assessment Program
NPS	National Park Service
NPSTORET	National Park Service Storage and Retrieval Database
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
Reference standard	Independently created solution of known concentration.
RPD	Relative Percent Difference
SDS	Safety Data Sheet

SOP	Standard Operating Procedure
STORET	Storage and Retrieval Database
USGS	United States Geological Survey
µg/L	Micrograms per liter
WRD	Water Resources Division of the National Park Service

12.4 Personnel Responsibilities

Personnel will study this SOP prior to beginning work on the project and follow its procedures in order to conduct the project according to outlined QA/QC procedures. This will ensure consistency and comparability when changes in personnel occur.

The role of the **project manager** is to serve as a liaison among other related water quality monitoring projects conducted by partners (e.g., state monitoring programs), park staff, other network staff (field personnel, data manager), contracted analytical laboratories, and other GLKN monitoring project managers. The individual will coordinate with resource management staff at the parks to ensure parks are informed of monitoring activities. Specific responsibilities of the project manager include the following:

- Coordinate field schedules and availability of supplies with field personnel
- Develop a training program for field personnel
- Develop, document, and oversee the implementation of standard procedures for field data collection and data handling
- Coordinate logistics with park staff
- Develop quality assurance and quality control measures for the project, supervise staff training and conduct quality assurance checks of field sampling techniques at least once, mid-season, with each field crew
- Contract with an analytical laboratory for analysis of water samples, ensure lab results meet program needs (e.g., QA/QC procedures, meaningful minimum detection limits for low level strength waters, adequate reproducibility of replicate samples)
- Supervise or perform data entry, verification, and validation
- Summarize and analyze data, prepare reports
- Serve as the main point of contact concerning data content

The project manager will also work closely with the data manager in the following capacities:

- Complete project documentation in NPSTORET and Aquarius (describing who, what, where, when, why and how of a project)
- Develop data verification, validation, and certification measures for quality assurance

- Ensure staff are trained in the use of database software and quality assurance procedures
- Coordinate changes to the field data forms and the user interface for project databases
- Identify sensitive information that requires special consideration prior to distribution
- Manage the archival process to ensure regular archival of project documentation, original field data, databases, reports and summaries, and other products from the project
- Define how project data will be transformed from raw data into meaningful information and create or oversee data summary procedures to automate and standardize this process
- Establish meaningful liaisons with state counterparts to promote sharing of data on a timely basis

The **field crew leader** will be responsible for preparing supplies and equipment for field season; ordering needed supplies; making travel arrangements; maintaining sampling equipment; maintaining field vehicles; ensuring field personnel follow sampling protocols; preparing field data forms, chain of custody forms, and analytical service request forms for each site; notifying contract laboratory of planned sample shipment date; and providing project manager with field trip and check-in schedule.

Field personnel should be adequately experienced and/or trained in using field and water quality sampling equipment. This experience is best obtained through a combination of classroom and hands-on training, including pilot-testing equipment at a nearby water body. Personnel should be familiar with the instruction manuals for all equipment, particularly with regard to calibration and maintenance procedures. See SOP #2, Training and Safety, for more details on training.

Field personnel will be responsible for following protocols and standard operating procedures during field activities and the handling of samples.

Roles of staff are explained in more depth in section 5 of the inland lakes protocol narrative, along with training and requirements.

12.5 Sampling Process and Design

The process of developing the sampling for this project included consideration of random versus nonrandom selection of lakes, and the sampling designs of Minnesota, Wisconsin, and Michigan. These three states have the greatest number of lakes of the lower 48 states, have active water quality monitoring programs and contain several of the network parks. Consistency of the network's monitoring design and protocol with neighboring state programs is desirable to facilitate data comparisons and allow statistical inferences using regional data. See the Inland Lakes Protocol Narrative for more information on these considerations.

Answering questions about all lakes within a park or all lakes across the network requires either a complete census of lakes or a random selection of lakes, which allows inference to the population of lakes as a whole. A complete census of lakes is not feasible, as the network contains well over 1,000 lakes. A random selection of lakes is not desirable because many lakes are inaccessible and would require more than a day of off-trail, backcountry travel to reach. We selected lakes, called index

lakes, at six park units within the network to span gradients of chemical and physical parameters, visitor use, watershed size, and spatial distribution within each park. The number of lakes selected at each park unit varies from one to nine (Table 1). We will analyze data separately for individual lakes and address questions at broader spatial extents through comparisons of trends across lakes. (See section 4 of the protocol narrative and SOP #9, Data Analysis, for more details).

Table 1. Number of lakes selected for long-term monitoring of water quality in each GLKN park unit.

Park Unit	Number of Lakes
Apostle Islands National Lakeshore	1
Indiana Dunes National Park	1
Isle Royale National Park	9
Pictured Rocks National Lakeshore	6
Sleeping Bear Dunes National Lakeshore	6
Voyageurs National Park	9

The information goals and statistical requirements determine the sampling frequency. To identify the long-term trends in a waterbody, the sampling frequency should be sufficient to identify a statistical trend beyond the background variability. Therefore, it is essential to evaluate existing data prior to establishing 1) the number of sites and 2) the frequency of sampling. Statistical power analysis based on the power to detect change guided the selection of these two parameters, using calculations of statistical sample size and analysis of sensitivity to sampling frequency, respectively. The goal is to be able to detect 20% change at 80% power and 5% significance. (See the inland lakes protocol narrative for more details on this power analysis.)

Sampling frequency, therefore, requires a pragmatic compromise among statistical rigor, logistics, and cost. For discrete monitoring the network will visit each selected lake three times, annually, during the open water season. Analyses of existing data, such as described in the protocol narrative, help us understand the limitations of our sampling design. We do not expect to be able to detect changes in most variables after only a few sampling years, and realize that it may take many years to detect changes in some highly variable parameters. Given our sampling frequency, we expect even the least variable parameter to require more than 10 years of monitoring data before we will be able to detect a 20% change with 80% power.

12.5.1 Site Selection

The lakes selected for sampling are listed in section 2 of the inland lakes protocol narrative. A single sampling site, typically located in the deepest part of the lake, will be the routine location for measuring all water quality variables. Sampling the deepest part of the lake allows sampling every possible depth to the bottom, and has a long history in limnology.

For discrete monitoring the network will use a 0–2 m integrating tube sampler, following the protocol used by Wisconsin and Minnesota and many other states. A near-bottom sample (approximately 1 meter from bottom) will be collected via Van Dorn sampler during mid-summer,

when lakes are stratified, for analysis of TP. Continuous water temperature profile data will be collected from a subset of index lakes across four parks, and co-located with discrete water quality monitoring locations.

12.5.2 Field Collection Parameters

Core field parameters will be measured with multiprobes or individual sensors (Table 2). Samples will be taken for shipment to a contract laboratory for nutrients (TP, TN, NO₃+NO₂-N, NH₄-N) and chlorophyll-*a* each sampling visit, or three times annually, and the remaining parameters (alkalinity, major ions (Cl, SO₄, Ca, Na, K, and Mg), DOC, and SiO₂) once annually. In addition, a near-bottom sample for TP will be collected once annually from thermally stratified lakes.

Collect water sample(s) with an integrated sampling tube or Van Dorn and process as per SOP #7. In the field log book and on the field data sheet, record information related to the sample collection, including:

1. Lake name and site identification code
2. Sample date, time, and depth.
3. The amount of water collected.
4. Whether duplicate samples for quality control were collected at this site.
5. Any additional notes or observations pertinent to this sample or location for this sampling period.

Additionally, always keep in mind the following:

- Sample containers should be labeled in indelible ink with, at a minimum, the station name, date and time of collection, and preservation method, if applicable.
- Follow all SOPs for sample collection and preservation.
- To ensure the integrity of the sample, be aware of possible sources of contamination. Contamination introduced during each phase of sample collection and processing is additive and usually is substantially greater than contamination introduced elsewhere in the sample handling and analysis process.
- Use appropriate procedures and quality-assurance measures that ensure sample representativeness and integrity and that meet study criteria. The degree to which a sample can be considered representative of a water body depends on many interrelated factors including temporal and spatial homogeneity of the water body, sample size, and the method and manner of sample collection.

12.5.3 Field Analysis

For discrete monitoring dissolved oxygen, temperature, specific conductance, and pH will be measured in the field using a multiprobe and following the methods listed in Table 2. Multiprobes typically perform within the specifications detailed in Table 3. Continuous temperature monitoring will also be conducted at select lakes, with the temperature sensors also in accordance with methods and specifications in Tables 2 and 3, respectively. Other parameters may be analyzed at the field station by field personnel in the future.

Table 2. Core suite of field variables and required *in situ* measurement method.

Field Variable	Method
Temperature (°C)	EPA 170.1
pH	EPA 150.1
Specific conductivity (µS/cm)	EPA 120.1
Dissolved oxygen (mg/L)	EPA 360.1
Clarity (cm)	Transparency tube, Secchi disk

Specification of quantification ability for field multiprobes and sensors is not a straightforward data quality objective exercise. Most field parameters tend to be those that characterize the waterbody and are not usually based on a criterion limit as would be used for a toxic pollutant. Therefore, instrument selection should be based on the parameters and ranges they can measure, but instrument-specific estimations of the range of uncertainty for each parameter will have to be made when interpreting data. Details on estimating instrument sensitivity are included below in section 12.8.2.

Table 3. Typical sensor performance specifications for multiprobe field instruments.

Sensor	Expected Range	Reporting Resolution*	Estimated Bias	Stabilization Criteria
Temperature	-5°C to 45°C	0.01°C	±0.15°C	Thermistor: ±0.2°C Glass: ±0.5°C
Specific Conductivity (SC25)	0 to 2000 µS/cm	µS/cm (range dependent)	±0.5% of reading +1 µS/cm	≤100 µS/cm: ±5% >100 µS/cm: ±3%
pH	1 to 14 units	0.01 unit	±0.2 units	±0.1 standard unit
Dissolved Oxygen (Conc.)	0 to 50 mg/L	0.01 mg/L	0 to 20 mg/L: ±0.2 mg/L 20 to 50 mg/L: ±0.6 mg/L	±0.3 mg/L
Dissolved Oxygen (% sat.)	0%–200%	0.1%	ca. ±2%	±2%
Depth – Z (pressure sensor)	0→100 m	0.1 m	ca. 0.1 m	0.1 m

* Resolution specifications are supplied by the manufacturers of the measuring meters. They are not necessarily closely related to real-world (outdoor) precision or bias and are sometimes more related to the number of significant figures reported rather than how accurate the extra significant figures are. This is why we will control measurement sensitivity in the actual outdoor measuring environment at least once a year by calculating alternative measurement sensitivity (AMS; see Irwin 2006 for more details on AMS).

12.5.4 Laboratory Analysis

Samples will be collected in the field according to SOP #6 for shipment to contract laboratories that meet QA/QC requirements outlined in Section 12.8.3 of this SOP. Samples will be analyzed for nutrients (TP, TN, NO₃/NO₂-N, NH₄-N) and total chlorophyll-*a*, each sampling period, while alkalinity, major ions (Cl, SO₄, Ca, Na, K, and Mg), DOC, and SiO₂, which tend to be less variable, will be monitored annually.

Methods chosen will meet quantification limits according to the criteria tabulated below (Table 4). The method limit of quantitation (ML) of the chosen method should be two to ten times lower than the typical expected low value. The ML is the lowest value that can be quantified with certainty. The value in the “ML needed” column of Table 4 is two to ten times lower than the lowest value found in the pilot year of sampling water quality for each analyte. This estimation is used as guide to the selection of the method that will be needed for this project.

The majority of the parameters listed in Table 4 are used in waterbody characterization and do not have criterion lower limits as would a toxic pollutant. Some of the criteria available are listed as maximums not to be exceeded and, as such, do not guide the selection of a method by ML. Guidelines to be used, therefore, include relevant state water quality standards, EPA eco-regional nutrient recommendations (USEPA 2000), state nutrient criteria as they are developed, data results for these parameters from the pilot studies carried out by GLKN, lowest values for the parameter found in relevant Horizon reports (NPS 1995a, 1995b, 1995c, and 1999), and/or values based on the best professional judgment of GLKN staff.

Table 4. Analytical detection levels required for GLKN water quality monitoring.

Analyte	Typical Low Value Found	Criteria Source ^{a, b}	Comment	ML Needed
Alkalinity (mg/L) ^c	ND (<1)	Lowest value pilot year	MDL unknown	
DOC (mg/L) ^c	3	Lowest value pilot year	Lab MDL 0.1	0.3 mg/L
Ca ²⁺ (mg/L) ^c	2.4	Horizon report	MDL unknown	0.24 mg/L
Cl ⁻ (mg/L) ^c	0.273	Lowest value pilot year	Lab MDL 0.025	0.02 mg/L
Chl- <i>a</i> (µg/L)	0.63	EPA	Lower of VII and VIII	0.06 µg/L
Mg ²⁺ (mg/L) ^c	0.47	Horizon report	MDL unknown	0.04 mg/L
K ⁺ (mg/L) ^c	0.24	Lowest value pilot year	Lab MDL 0.06	0.02 mg/L
Na ⁺ (mg/L) ^c	0.5	Horizon report	MDL unknown	0.05 mg/L
SO ₄ ²⁻ (mg/L) ^c	0.625	Lowest value pilot year	Lab MDL 0.025	0.06 mg/L

TP (µg/L)	10	EPA	Lower of VII and VIII	1.0 µg/L
TN (µg/L)	0.38	EPA	Lower of VII and VIII	0.03 mg/L
NH ₄ -N (µg/L) ^c	5.0	Horizon report	MDL unknown	1.0 µg/L
NO ₃ +NO ₂ -N (µg/L) ^c	2.0	Horizon report	MDL unknown	1.0 µg/L
SiO ₂ (mg/L)	ND (<0.1)	Lowest value pilot year	Lab MDL 0.2	0.1 mg/L

NOTES:

a: EPA = EPA Ecoregional nutrient criteria recommendations, lower value recommended for aggregate ecoregions VII and VIII (USEPA 2000).

b: WQS = criteria value listed in Ledder (2003) state water quality standards

c: parameter has no official criterion for any of the states, the value reported in the analytical requirement column is the lowest value determined in the pilot year for rivers or lakes or the lowest value found in a spot check of Horizon reports for each park.

12.6 Sampling Methods

Consistent methods are important to long-term quality data. In actuality, the ideal conditions are not always met in the field or in the lab and changes in staff do occur. Therefore, documentation of procedures, site conditions, laboratory analysis, and reasons for deviations of any kind is important in and of itself for long-term projects. Personnel will be encouraged to write down more than they feel may be necessary in the moment as the future interpretation of their data will depend on the written record and not the memory of an individual.

12.6.1 Site Locations

Refer to description of monitoring station location, directions, and photos to verify correct location. Verify coordinates with a GPS unit. Document this verification. This information will be contained in a site binder along with a table of previous field measurements to compare with new measurements.

A single sampling site, typically located in the deepest part of the lake, will be the routine location for measuring all water quality variables. Sampling the deepest part of the lake allows sampling every possible depth to the bottom and has a long history in limnology. Except for shallow lakes, this type of sampling ignores the littoral zone and always avoids the nearshore zone, as well as embayments and other features related to morphometry.

12.6.2 Field Water Measurements

Before making field measurements during discrete monitoring, properly calibrated sensors (see SOP #6) must be allowed to equilibrate to the condition of the water being monitored. Sensors have equilibrated adequately when instrument readings have stabilized, that is, when the variability among measurements does not exceed an established criterion. The criteria for stabilized field readings were defined operationally by O’Ney (2005) for a set of three or more sequential measurements (Table 5).

The natural variability inherent in surface water or ground water at the time of sampling generally can be compared with these stability criteria and indirectly relates to the short term bias or the long term accuracy that should be attainable with a calibrated instrument. Dissolved oxygen sensors that utilize a Clark cell membrane may require up to five minutes to stabilize, while optical dissolved oxygen sensors and sensors for the other parameters typically require less than 60 seconds to stabilize. Observers should only note instrument readings after the stabilization criteria in Table 5 are met for all the sensors, and then record readings for all parameters at once.

Measure field water quality variables and conduct sampling according to SOP #6, field measurements and water sample collection. Quality results require consistent measurement methods and detection limits.

Depth profiles of temperature, specific conductance, pH, and dissolved oxygen will be measured at each sampling station using a multiparameter instrument (multiprobe). Lake level will be determined at benchmark stations on a regular basis. Details of methods for measuring lake levels are included in SOP #4. Clarity will be measured using a Secchi disk or transparency tube. These core parameters will be measured when water samples for analysis of the advanced parameters are collected.

Begin just below the water's surface (ca. 0.3 m depth) and take readings after stabilization of the multiprobe. Lower the sensors to collect a vertical profile of field parameters at 1m intervals until 20 m depth, and then every 2–5 m depending on overall depth and gradients. Replicate 10% of the readings (e.g., at 1 m, 10 m, etc.); take the replicate readings immediately following the original readings. Values should agree within 10% or the acceptance criteria in Table 5, whichever is larger.

Table 5. Recommended instrument stabilization criteria for recording field measurements^{a,b}.

Standard Direct Field Measurement	Stabilization Criteria (O'Ney 2005)	Stabilization Criteria <i>In situ</i> Multisensors (WOW 2005)
Temperature^c:		
Thermistor Thermometer	± 0.2°C	± 0.2°C (5%)
Liquid-in-glass Thermometer	± 0.5°C	
Specific Conductivity (SC25)^d		
When ≤ 100 µS/cm	± 5%	<5 µS/cm (10%)
When > 100 µS/cm	± 3%	
pH^e: Meter displays to 0.01	± 0.1 unit	± 0.2 unit (10%)
Dissolved oxygen^e:		
Amperometric (same as polarigraphic) method	± 0.3 mg/L	± 0.5 mg/L (10%)

a: Resolution/sensitivity is a data quality indicator related to detection limits but typically handled differently for field probes than for laboratory parameters. For more information, see Irwin (2004).

b: In the case of field probes, bias is typically a best-case maximum deviation from known correct values (typically based on comparisons with known NIST certified reference materials or standards). True accuracy is a

combination of high precision and low bias and is hard to quantify with the small sample sizes used to control bias and precision in typical field measurements (see Irwin 2004 for more details).

c: Recommended sensor calibration is quarterly.

d: Recommended sensor calibration is daily.

e: Recommended sensor calibration is at beginning of sampling day with a calibration check at the end of the day.

Sensors that record water temperatures at 1-hour intervals year-round from the surface to bottom at 1-meter intervals will be deployed in a subset of index lakes (see SOP 14 for details). Since the sensors are simply deployed with no connection to a data logger, assessment of sensor stabilization occurs after data are downloaded.

At all sites, record visual observation information required on the data sheet. Such observational data can provide important information to the interpretation of field measurements.

If any analyses are to be done in-house, conduct these analyses as soon as possible upon return from the field. A clean analytical station should be prepared in which to work, free of food items, mud, lubricants, or lab chemicals. Hands should be thoroughly washed.

12.6.3 Samples for Laboratory Analysis

Prepare bottles and labels prior to field sampling as per SOP #6. Collect samples using a 0–2 m integrating tube sampler as per SOP #6. For all sampling, it is critical to avoid sampling water showing evidence of oil, gasoline or anything else from the boat. It is best to kill the engine and set the anchor, if possible, although this may not be possible or advisable in bad weather or with a balky engine.

Prior to filling sample bottles sent by the laboratory, first rinse the bottle once with sample water, if the bottle is not pre-preserved. Some analyses require preservation of the sample with acid, which may be added to the bottle by the contract laboratory; in these cases do not rinse the bottle first. Take care not to overfill the bottle if the bottle is pre-acidified, as overfilling will flood the acid out of the bottle. If the samples requiring acid preservation are not pre-preserved, use caution to add the ampule of preservative supplied by the lab and immediately rinse hands in water if acid is spilled. Protective gloves are recommended.

Table 6 summarizes the variety of methods, detection limits, preservation techniques, and holding times for water samples addressed by this protocol. These methods conform to those used by Minnesota, Wisconsin, and Michigan for state certification of environmental laboratories involved in Clean Water Act or drinking water sample analysis (MDH 2005, WSLH 2003, MDEQ 2005). They are also used by EPA-funded research projects of natural waters in the upper Midwestern U.S. Holding times shall in no case be less stringent than those recommended by EPA in 40 CFR Part 136 to 136.3 and appendices. Refer to SOP #6 for additional details regarding sample collection and preservation.

Table 6. Example range of analytical methods, method detection limits (MDLs), containers, and holding times.

Analyte	Analytical <i>Note 1</i>	Method #	Det. Limit	Vol. (ml)	Filter	Preservation	Sample Bottle <i>Note 2</i>	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		<i>Note 4</i>	None		None
Calcium	ICP	3120B APHA	10 ug/L		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
	Titrimetry	215.2 EPA-NERL	0.5 mg/L		<i>Note 3</i>	4°C		6 mo
	FAA	I-3152 USGS-NWQL	0.1 mg/L	250 mL	<i>Note 3</i>	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-CI APHA	0.15 mg/L	100 mL		4°C	P or G	28 d
Chlorophyll-a	Spect.	10200 APHA	2 ug/L	≤1 L	<i>Note 4</i>	Freeze filter	P	30 d
Color	Spect.	110.2 EPA-NERL	5 Pt units		<i>Note 5</i>	4°C	G	48 hr
	Vis. Comp.	I-1250 USGS-NWQL	1 Pt-co	250 mL	<i>Note 5</i>	4°C	P	30 d
DOC	Spect.	415.3 EPA	0.018 mg/L	125	<i>Note 3</i>	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		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	5 ug/L		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
Mg	ICP	3120B APHA	20 ug/L		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	0.5 ug/L		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
Na	ICP	3120B APHA	30 ug/L		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
	FAA	3111B APHA	2 ug/L		<i>Note 3</i>	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		24hr / 28d
	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		<i>Note 3</i>	pH<2 HNO ₃	P or G	6 mo
	Spect.	4500- SiO ₂ D APHA	0.04 mg/L		<i>Note 3</i>	No, 4°C	P	28 d
	FIA-Spect.	4500- SiO ₂ F APHA	0.78 ug/L		<i>Note 3</i>	No, 4°C	P	28 d

Standard Operating Procedure #12: Quality Assurance/Quality Control

TSS	Gravimetric	I-3765 USGS-NWQL	1 mg/L	250–500	Note 4	4°C filter	P	NA
SO ₄	IC	4110C APHA	75 ug/L		Note 3	pH<4 H ₂ SO ₄	P or G	
	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.

Samples are stored in a cooler with ice packs during field sampling. Prepare samples for shipment according to the contract laboratory's protocols. These protocols will be provided to the field sampling personnel for each sampling round so that the proper procedures are accessible in the event of contract laboratory changes. In general, samples are shipped on ice to maintain a temperature of approximately 4°C. A plastic bag is placed in the cooler first. Sample bottles are packed among zip lock bags of ice and/or ice packs to prevent water leakage into the sample bottle during shipment. 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. Some analyses require the sample to be frozen for shipment; such frozen samples are likewise packed among ice packs for shipment to the contract laboratory.

Follow the shipping company's requirements in preparation of the cooler of samples for shipment. Packaging problems may cause delays in shipment, which can mean that samples do not arrive at the laboratory at the proper temperature or past holding time, compromising data quality. In general, the ice and samples should be contained within a sealed plastic bag within the cooler so that the cooler does not leak. Packing tape should be wrapped around the cooler vertically and around the lid seam horizontally to ensure it remains closed. A note on the cooler that it contains water samples is helpful as well so that if leakage occurs, carriers will know the contents are not hazardous.

12.6.4 Sampling Forms

Before leaving the monitoring site, all field forms and sample labels must be reviewed for legibility, accuracy, and completeness. Any changes in procedure due to field condition must be explained in the comments section. Make sure the information is complete on all forms. Record the departure time on the field form. After reviewing each form, initial the upper right corner of each page of the form. Document any photos taken by including the photo number and roll number or digital camera photo number on the field form.

12.7 Handling and Custody

One part of proper data and sample handling procedures is to provide a complete record of the methods and procedures followed. Complete records are important to long-term monitoring so that anyone using the data may trace the sampling history.

12.7.1 Field Data

Field data will be collected on forms printed on waterproof paper. While at each monitoring site, the information recorded on the forms should include:

- Date and day of week
- Time of arrival
- Names of field team members
- GPS coordinates, to verify location

- Current weather (air temperature and wind speed) and relevant notes about recent weather (storms or drought)
- Observations of water quality conditions
- Description of any photographs taken
- Multiparameter sonde (model), calibration date, and field measurements of core suite variables
- Sample identification numbers and collection times for advanced suite variables or quality assurance samples
- Samples taken for laboratory analysis
- Whether any samples were not collected, and reason
- Water level
- Any other required metadata for NPSTORET or Aquarius data entry
- Time of departure

All entries should be made clearly. If an incorrect entry is made, a single heavy line should be drawn through the incorrect entry and the correction made. All corrections should be initialed and dated. The completed field forms will be maintained in chronological order by station, copied into site binders and the originals maintained on file indefinitely. Field data are reviewed annually by network personnel (see SOP #8, Data Entry and Management, for details).

12.7.2 Site/Sample Identity Codes

Sampling stations will be identified by park and water body according to network guidance. Information on the sampling station and park will comply with NPSTORET and Aquarius requirements.

12.7.3 Data Transfer

Import field and laboratory data into NPSTORET or Aquarius as soon as possible after receiving the data, according to SOP #8. Field forms are the only written records of field measurements; place copies in office binders and keep originals on file indefinitely. Program sampling data and associated records are archived and stored in the GLKN Ashland Office. Boxes are numbered consecutively by year, project, and station number.

Personnel importing data into the database should take care to ensure that units are consistent for laboratory data. Different laboratories may report analytical results in different units (mg/L vs. µg/L) for the same analyte. Follow SOP #8, Data Entry and Management, when importing data.

Contract laboratories should use the STORET-supported laboratory remark codes (Table 7) or provide a map relating their remark codes to these. Unlisted remark codes should be discussed for possible addition by the USEPA to the STORET codes. The detection descriptors to be used in data entry to STORET are listed in Table 8.

Table 7. STORET-supported laboratory remark codes.

Acronym	Remarks
AL	Aldol condensation present. Analyte may not be present.
CNT	Non-acceptable colony counts.
EHT	Sample or extract held beyond acceptable holding time.
FBK	Analyte found in blank. Sample contamination indicated.
FDB	Failed. Dry blank not acceptable.
FDC	Failed. Drift check not acceptable.
FIS	Failed. Internal standard not acceptable.
FLD	Failed. Lab duplicate not acceptable.
FFD	Failed. Field duplicate not acceptable.
FFB	Failed. Field blank not acceptable.
FFS	Failed. Field spike not acceptable.
FFT	Failed. Trip blank not acceptable.
FLC	Failed. Linearity check did not meet quality criterion.
FLS	Failed. Lab spike recovery not acceptable.
FMS	Failed. Matrix spike recovery not acceptable.
FPC	Failed. Lab performance check not acceptable.
FQC	Failed. Quality control criteria exceeded during analysis.
FRS	Failed. Internal reference sample not acceptable.
FSP	Failed. Surrogate spike recovery not acceptable.
FSB	Failed. Spiked field blank recovery not acceptable.
FSL	Failed. Spiked lab blank recovery not acceptable.
INT	Interference suspected. Analyte may not be present.
ISP	Improper sample preservation noted. Analysis performed.
LIS	Lab internal standard(s) added to sample.
LLS	Value less than lower quality control standard.
PRE	Presumptive evidence that analyte is present.
NJ	TIC, Tentatively Identified Compound, result is approximate
N	TIC, Tentatively Identified Compound, presumptive id only
OUT	Result value is defined as an outlier by data owner
SUS	Result value is defined as suspect by data owner

Table 8. STORET detection descriptors.

Detected and Quantified
*Non-detect
*Present >QL
*Present <QL
*Not Reported
*Present

12.7.4 Sample Transfer

Conduct sample processing per SOP #7. Refrigerate or freeze samples, as required. Conduct in-house laboratory work and package samples for sending to contract analytical laboratory. Sample preservation and conditions for shipment will differ for each parameter; these needs should be discussed ahead of time with the contract laboratory and documented. Fill out the analytical request form and chain-of-custody (COC) form provided by the laboratory (examples are included in SOP #7). The COC form is used to document the taking, shipment, and receipt of samples. The laboratory will use the COC to check samples into the analytical process. Clean all transfer bottles and equipment according to SOP #7. Water samples are maintained as close to sampling conditions as possible by shipping on ice. Chain-of-custody documentation will be maintained. A chain-of-custody includes not only the form, but all references to the sample in any form, document, or log book that allow tracing the sample back to its collection, and documents the possession of the samples from the time they were collected until the sample analytical results are received.

12.8 Analytical Methods

Field equipment and contract laboratories are likely to change during the course of this long-term monitoring project. Documentation will be maintained, therefore, as regards each multiprobe and contract laboratory's ability to meet the data quality objectives of this project. Forms for this purpose are included in Attachment A. These completed forms for each multiprobe and contract laboratory will be maintained along with the maintenance logs for the multiprobe and QAPPs for each analytical laboratory.

12.8.1 Field Methods

One important aspect in the low uncertainty/bias and high precision of a water quality monitoring program is the correct selection of probes for measuring field variable and their subsequent calibration and maintenance. Table 3 lists typical field sensor performance specifications that should be expected from monitoring equipment for this protocol. Table 9 summarizes the ideal calibration frequency and minimum acceptance criteria for these sensor probes. The reality of logistical constraints at back country sites may preclude calibration and checks of calibration at the ideal frequency.

Table 9. Ideal calibration frequencies and acceptance criteria for field instruments.

Parameter	USEPA Method	Minimum Calibration Frequency and QC checks	Acceptance Criteria	Corrective Actions
Temperature	170.1	Annually, 2-point check with NIST thermometer	$\pm 1.0^{\circ}\text{C}$	Re-test with a different thermometer; repeat measurement
Specific Conductance (SC25)	120.1	Daily, prior to field mobilization; calibration check prior to each round of sampling; 10% of the readings taken each day must be duplicated or a minimum of 1 reading if fewer than 10 samples are read.	$\pm 5\%$ RPD 10%	Re-test; check low battery indicator; use a different meter; use different standards; repeat measurement
pH	150.1	Daily, prior to field mobilization (two buffers should be selected that bracket the anticipated pH of the water body to be sampled)	± 0.05 pH unit	Re-test; check low battery indicator; use different standards; repeat measurement; don't move cords or cause friction/static
		Calibration check w/ third buffer prior to each round of sampling; check with low ionic strength buffer in addition, if conductivity is $< 50 \mu\text{S/cm}$	± 0.1 pH unit	
		10% of the readings taken each day must be duplicated or a minimum of 1 reading if fewer than 10 samples are read.	RPD 10%	
Dissolved Oxygen	360.1	Daily, prior to field mobilization; check at the field site if elevation or barometric pressure changed since calibration	0.2 mg/L concentration or $\pm 10\%$ saturation	Re-enter altitude; re-test; check low battery indicator; check membrane for wrinkles, tears or air bubbles; replace membrane; use a different meter; repeat measurement; allow more time for stabilization
Depth	--	Daily, prior to field mobilization, check at the field site. Check annually against commercially purchased brass sash chain labeled every 0.5 m to ensure that it reads zero at the surface and varies < 0.3 m for depths < 10 m and no more than 2% for greater depths.	± 0.1 m	Retest, check low battery indicator; repeat measurement; use with accurately calibrated line
Transparency tube	--	Transparency tubes have a 100 or 120 cm scale; ensure tube is clean	± 1.0 cm for transparency tube	
Marked lines (e.g., Secchi, Van Dorn)	--	Check markings annually against brass sash chain. If lines are heated (for decontamination) check prior to each round of sampling.	$\pm 1\%$, 0–10 m $\pm 2\%$, > 10 m	Re-mark line.

Ensure calibration standards are not used beyond expiration dates. Refer to SOP #6 for an explanation of each parameter measured, guidelines on potential field measurement problems, and other details on performing calibration checks. Keep the manufacturer's manual with each instrument for aid in troubleshooting.

Used calibration solutions, in general, may be rinsed down a sink with water after consideration of the wastewater treatment system available to that sink. Safety data sheets (SDS) which are sent with manufacturer purchased calibration solutions should be kept on file. These documents describe the flammability, toxicity, and other safety hazards of reagents. Some reagents may include constituents toxic to aquatic life. These should not be rinsed down a sink in any large quantities in primitive areas where the ultimate destination of wastewater is the aquatic environment. These reagents should be collected in a leak-proof container that is properly marked and disposed of in an adequate treatment system.

Calibration logs for multi-parameter sondes will be maintained and will document the frequency of calibration and calibration checks. Data reporting units will be standardized by using the data sheet. Keep calibration and maintenance logs for multiparameter sondes with the sonde during the sampling season. Logs will later be archived at the network office in Ashland, Wisconsin. A new log will be started for each field season.

12.8.2 Field Equipment Performance Criteria

Estimates of the completeness, representativeness, precision, comparability, and systematic error of data generated by the use of a multiprobe will be estimated and documented according to this SOP.

Data will be considered representative of the lake when procedures detailed in the inland lakes protocol narrative and associated SOPs are followed. Actual sampling location and changes due to field conditions will be documented on the field forms. Data will be comparable year-to-year as objectives and methods chosen are consistent within documented NPS GLKN sampling procedures.

12.8.2.1 Field Data Completeness

The completeness of data collected, or percentage of intended field measurements that were actually made, will be calculated at year's end according to the formula below.

Equation 1:

$$\text{Percent_completeness} = \left(\frac{\# \text{ samples_collected}}{\# \text{ samples_planned}} \right) \times 100$$

12.8.2.2 Instrument Sensitivity

Sensitivity of the multiprobe will be documented through an estimation of the limits of detection known as alternative measurement sensitivity (AMS). The AMS for each multiprobe will be estimated annually, and for any new multiprobes, by taking multiple (at least seven) measurements in a field simulation for each parameter. Alternative Measurement Sensitivity (AMS) is a two-sided estimation based on the 99% confidence interval for sample size seven. Of interest is the size of the difference between two individual values that can be considered to actually be a true difference. This

calculation differs from the MDL, which is a one-sided true difference from zero. The standard deviation will then be calculated and used in the estimation of AMS ($3.708 \times \text{SD}$). This estimation will be carried out and documentation kept for a new probe and annually thereafter (Attachment A). Documentation will be kept in the project files.

12.8.2.3 Instrument Precision

The precision of the multiprobe will be estimated through the use of duplicates. Duplicates will be measured each day in the field at a rate of 10% (or 1 per park sampled if fewer than 10 sites are sampled). A reading will be considered a duplicate when it is repeated at a particular sampling site immediately after it is initially taken. Only the surface reading need be replicated. This will reduce the contribution of variability from the variability of the lake itself. Precision calculations are made by way of a Relative Percent Difference (RPD). Measurement Quality Objective (MQO) for precision: The RPD should be less than or equal to 10%; details and corrective actions are listed in Table 10. Calculate precision as in Equation 2.

Equation 2:

$$\text{Precision} = \left(\frac{A - B}{(A + B) / 2} \right) \times 100$$

Where A is the first reading, and B is the second reading taken immediately after the first.

Table 10. Frequency, acceptable range, and corrective actions for quality control (QC) samples.

Type of QC Sample	Frequency	Acceptable Range for MQOs	Corrective Action
Field Duplicates (samples)	Minimum of 1 per trip per parameter or 10% of all samples per parameter per day	All parameters $\pm 15\%$ RPD, chlorophyll-a, TSS and nutrients $\pm 30\%$	Audit field personnel and verify sample collection procedure; resample; reanalyze; revise SOP; audit and train field personnel; project manager determines whether associated data is usable
Field Duplicates (multi-probes)	Minimum of 1 per trip per parameter or 10% of all samples per parameter per day	All parameters $\pm 10\%$ RPD	Re-calibrate instrument; replace batteries; perform instrument field check with different standards; repair or replace instrument; notify management; audit and train field personnel; project manager determines whether
Laboratory Control QC (bias)	One each per analytical batch, minimum 1 per 20 samples	All parameters $\pm 15\%$ RPD	According to laboratory QAPP, ensure re-calibration, re-analysis, and documentation.

12.8.2.4 Instrument Bias

The systematic error/bias of the multiprobe will be estimated through the use of reference solutions. Reference solutions will be measured prior to each round of sampling, in the lab or field. The results

will be documented and used as a calibration check according to Table 9 as well as for a long-term estimate of systematic error/bias. Bias calculations are made by way of a Percent Difference (PD). Calculate bias as in Equation 3.

Equation 3:

$$Bias = \left(\frac{Y - X}{X} \right) \times 100$$

Where X is the known (or expected) amount, and Y is the measured concentration.

12.8.3 Laboratory

The ability to use environmental data to reveal long-term trends requires consistent analytical methods and detection limits. The NPS recommendation is to use only laboratories with NELAC certification or at least certification by the state programs that also use the laboratory. The chosen contract laboratories must prove their capability annually through participation in blind quality control checks and other methods prescribed by the states in which they receive certification and/or federal programs in which they participate. Copies of certifications for each analyte and/or method will be kept on file along with Quality Assurance Project Plans (QAPPs) for each laboratory contracted for the duration of this monitoring effort.

The method used in calculating method detection limits (MDLs) and method limit of quantitation (ML) or laboratory reporting limits (Lt-RL for USGS labs) may differ for each contract laboratory. Most laboratories routinely recalculate MDLs, MLs, and QC sample control limits using repeated measurement of standard samples or multiple percent recoveries on a quarterly or annual basis. The GLKN will request and maintain copies of this information as most recently calculated for the relevant analytical methods to assist in the selection of contract laboratories, data validation, and AMS calculations.

A checklist to be used in selecting contract laboratories and documenting their compliance with GLKN QA/QC expectations is included in Attachment A.

Great Lakes Network staff validating laboratory data for database entry should take care to ensure that data are entered in consistent units, as different laboratories may report results in different units (mg/L or µg/L) for the same analytes. A checklist to be used in data validation is included in Attachment B.

12.8.4 Laboratory Performance Criteria

Comparability, representativeness, precision, systematic error, and completeness of data generated by contract laboratories will be estimated and documented according to this SOP during data validation. Table 10 can be used as a guide. Unless otherwise justified (for example, to be consistent with State requirements), in no case will Measurement Quality Objectives less stringent than the following be accepted:

- Precision—A maximum of 10% RPD for all lab parameters except chlorophyll-*a* and nutrients, for which the maximum RPD is 30%.
- Bias—A maximum of 15% PD for all parameters, or State credible data defaults, whichever is more stringent.

All MQOs will be used as data rejection criteria.

The data delivery package must contain QC sample results and an explanation of new STORET and NPSTORET compatible laboratory flags. A checklist to be used for documenting this procedure is included in Attachment B.

Data generated will be considered representative of the particular site when samples are taken according to the sampling protocol for each sampling objective. Actual sampling location and changes due to field conditions will be documented on the field forms. Data will be comparable year-to-year as objectives and methods chosen are consistent within documented sampling procedures.

12.8.4.1 Laboratory Data Completeness

The completeness of data analyzed, or the percentage of intended sample measurements that were actually made, will be calculated at year's end according to Equation 4. Data points may be missed due to site conditions, sample container breakage, or disqualified analyses due to control limit exceedances in the laboratory. The reasons for missing data should be documented.

Equation 4:

$$\text{Percent_completeness} = \left(\frac{\# \text{samples_analyzed}}{\# \text{samples_planned}} \right) \times 100$$

Determining required sample sizes and attendant completeness goals was done in a stepwise manner, considering desired statistical power and minimum detectable differences (see discussions in the protocol narrative). Should percent completeness ever fall below sample sizes needed for analysis of trends, adaptive changes will be made to ensure it does not happen again.

12.8.4.2 Laboratory Sensitivity

Measurement sensitivity is estimated in laboratory analysis through the use of signal to noise ratios or the standard deviation of repeat measurements of a low level reference standard. The method each laboratory uses to calculate MDLs and MLs will be documented using the data validation check list. This information will be reviewed during data validation and kept along with other QA/QC information for each laboratory.

12.8.4.3 Laboratory Precision

Measurement precision and bias are estimated using a number of QC samples during a round of sample analysis for each method. A reference standard of a certified known concentration is analyzed along with samples at a rate specified by the laboratory's QA/QC program. The results of these samples are reported and the percent difference calculated. Laboratory duplicates of field samples are also analyzed. This is a field sample that is split or subject to repeat analysis within the laboratory to

estimate and control precision repeatability. Relative percent differences are calculated for laboratory duplicates. The percent difference and RPD of these QC samples should fall within the control limits specified by the laboratory (usually 10% to 20% depending on the type of analysis made).

Field duplicates are to be sent to the analytical laboratory at a rate of at least one in every 10 samples. A field duplicate is a split sample, or at minimum, a sample co-located in time and space. The duplicate is usually given a separate sample identity on the sample label and forms, and it is noted on the field form for which sample this is a duplicate. This is a separate check on precision and repeatability of the laboratory analysis. The precision measurement is calculated using the Relative Percent Difference (RPD) between duplicate sample results per analyte. Precision estimates should be performed within seven days of receipt of laboratory results as part of data validation. Acceptance criteria and corrective actions are summarized in Table 10.

12.8.4.4 Laboratory Bias

Systematic error/bias (formerly referred to as percent recovery) is estimated for each laboratory method through the analysis of spiked samples and/or certified reference materials. Spiked sample analyses are conducted by the contract laboratory to ensure the reported data are accurate, or compare favorably to the true values. A spiked sample consists of a sample with a known concentration of analyte added before any sample preparation procedures are carried out. Sample and spiked sample are analyzed using normal procedures and the percent spike difference is calculated. Percent difference is calculated as bias (Equation 3).

Other sources of systematic error in monitoring programs include sample cross contamination from field sampling equipment used to handle a multiple number of samples. Equipment blanks are used to estimate whether systematic error is added to sample data during sample handling. The integrated sampler is to be rinsed in the field three times at each site prior to taking samples. Compositing jugs that are not site-dedicated may also be a source of cross contamination. Collect an equipment blank periodically, as follows:

1. In between sample sites, rinse the equipment used to transfer water samples (integrating sampler or compositing jug) with laboratory reagent-grade water three times and discard.
2. Rinse with a fourth aliquot and save this aliquot to lab bottles as if it were a lake sample to be analyzed.

This sample is labeled equipment blank and information kept on a datasheet describing the source of the blank. Results for all parameters should be non-detect. This type of blank is a check for cross contamination between sampling sites and control for bias introduced by cross contamination.

The laboratory QAPP should define control limits to be used during analysis of samples. If the analysis QC samples are not meeting the control limits, the analysis is usually repeated after re-calibration. This protocol should be documented in the lab's QAPP. During data validation GLKN will ensure that the laboratory performance meets the precision and bias MQOs tabulated in Table 10.

12.8.5 Changing Methods and Documenting Cumulative Bias

When a field method is changed, the cumulative bias, or change in sampling results due to the method change alone, should be estimated. Cumulative bias can become significant over time even though changes in methods are small. When change occurs due to a scheduled change in staff, both the new staff member and old staff member should perform side-by-side field measurements several times (minimum of seven) during training when possible. The results for both will be compared as below.

When purchasing a new probe, it should be used for a minimum of seven measurements side-by-side with the existing probe when possible.

When a change in laboratory methods is made by the laboratory, the laboratory will be responsible for estimating any bias introduced by the change. When a laboratory contract is changed to a different laboratory, a minimum of seven samples will be split for analysis in both labs. Data from these comparisons will be used to calculate a percent difference (PD, Equation 5) and fraction of change (Equation 6).

Equation 5:

$$PD = \left(\frac{new - old}{old} \right) \times 100$$

Equation 6:

$$Fraction_of_change = \left(\frac{old}{new} \right)$$

Where *old* = data from the older or original method and *new* = data from the new or replacement method.

Ideally, the average PD (two-sided 95% t-distribution confidence interval) will be within 20% of the mean of the old or the new method measurements. If this is not the case, the number of overlapping measures should be increased until this criterion is met (Irwin 2006). New values can then be normalized to the old method value by multiplying them by the fraction of change.

Any bias should be stated clearly and documented. Documentation will be stored within each site file and should also include:

- all raw data pairs for future use
- standard deviation
- the MDL or AMS of the method
- number of paired samples
- 95% t-distribution confidence interval for the measurement difference
- dates between which overlapping measurements were made
- date documentation calculation was made

This comparison of methods will be carried out, when possible, in order to document any bias that is introduced into the long-term data by a change in methodology and not in the actual environment. Due to the logistical realities imposed by sampling in remote locations and the reality of equipment loss or damage and abrupt changes in staff, this type of comparison may not always be possible.

12.9 Instrument Calibration Frequency, Inspection, and Maintenance

Instruments used in field measurement or laboratory analysis often require frequent inspection and maintenance to ensure they are in good working order. Calibration frequencies differ by instrument and should be spelled out in SOPs for both field and laboratory work to ensure consistency.

12.9.1 Field Instruments

Each instrument must have a logbook. The calibration schedule must be observed, using fresh calibration standards. Calibration solutions may be disposed of by rinsing down the work space sink if the contents do not include constituents which would harm the pipes or aquatic life on the receiving end of the available wastewater treatment (see relevant SDS sheets). Calibration checks must be documented.

When sensor probes are to be stored for extended periods of time, thoroughly clean sensors, remove batteries, and store sonde according to specific instructions in SOP #11 and manufacturer's manual. Store calibration standards and electrolyte solutions in a temperature-controlled environment.

12.9.2 Laboratory Instruments

Instrument calibration and verification is performed at least once a day for each analytical method prior to the analysis of samples. Each laboratory specifies its own procedure, which should include multiple point calibrations plus a blank, continuing calibration verification, and final calibration verification at the end of an analytical run. Each laboratory should define the procedures in a QAPP. Review of these procedures will be documented using the laboratory checklist in Attachment B.

12.10 Inspection and Acceptance of Supplies and Consumables

Monitoring water quality requires many pieces of equipment and a large amount of supplies. Table 11 lists the SOPs that include checklists of supplies and equipment. When new equipment is received or equipment is returned after repair, inspect it for flaws and test it to ensure proper functioning. When supplies are received, inspect them to ensure containers are properly sealed. Ensure reagent containers are dated upon receipt and upon opening. Commercially purchased calibration standards come with an expiration date that must be observed.

Table 11. Checklists of equipment and supplies for monitoring water quality of inland lakes.

Checklist	Location
Safety equipment and supplies	SOP #2
Decontamination equipment and supplies	SOP #5
Field equipment and supplies	SOP #6
Laboratory equipment and supplies	SOP #7

12.11 Records Management

All records must be kept, from field activity through sample results. Required metadata will be completed. Table 12 summarizes the QA/QC procedures related to data management. For more details on the Great Lakes Network's overall strategy for metadata generation, management, and distribution see chapter 8, Data Documentation, of GLKN's Data Management Plan (Hart and Gafvert 2006) and the appendices of that document.

Table 12. Summary of QA/QC procedures pertaining to data management.

Procedure	Description
Instrument calibration logs	Each instrument must have a logbook.
Field forms	Field forms are the only written record of field measurements. Place copies in site binders and keep originals on file indefinitely.
Estimating precision	The precision measurement is calculated using the Relative Percent Difference (RPD) between duplicate sample results per analyte. Precision estimates should be performed within 7 days of receipt of laboratory results.
Electronic data entry	Approximately 10% of electronic data entries should be spot checked for errors on a random basis. If errors are found, another 10% are spot checked.
Data archiving	Sampling data and associated records are archived in boxes and stored at the GLKN Ashland office. Boxes are numbered consecutively by year, project, and station number.
Data validation	Data validation is the process that determines whether quality control objectives for data collection were met.
Data validation reports	Data validation reports provide a narrative that discusses any deviations from QA/QC procedures and the impacts of those deviations.
Data verification	Data verification demonstrates that a data set will qualify as credible data.
Data verification reports	Data verification reports document the results of the data verification procedure.
Data qualification codes	Data must be fully qualified before uploading to the Water Resources Division's NPSTORET

Data will be archived according to SOP #8 (Data Entry and Management) for digital data, paper copies, and field forms. Field forms are maintained indefinitely. Brief characterizations of the data from each NPS unit that was sampled and the network as a whole will be performed each year, after all QA/QC procedures have been completed. For each station sampled, these descriptive statistics will include mean, median, maximum and minimum values, skew, kurtosis, and measures of variability (e.g., CV, standard error, variance) for each water quality variable. Understanding of landscape-scale dynamics will be provided by analyses of variability among stations within the domain of interest (e.g., park unit), following one of several approaches described in SOP #9, Data Analysis.

A ‘Memo-to-file’ will be used to document any decisions or corrections that are made. This memo will include the date, name of author, site or sample referred to, a description of the problem or error and a statement describing the decision made or action taken. The memo will be archived with the appropriate site data and files.

12.12 Assessment and Oversight

It is the project manager’s responsibility to make sure each component of this and other SOPs are followed.

12.12.1 Corrective Responsive Actions

The project manager, in consultation with experienced professionals, will be responsible for taking corrective responsive action in the case in which QA/QC is not followed or in the case of an unexpected event. Responsive action is often needed in the event of broken sample bottles, missing data, errors on field sheets, changes due to field conditions, problematic analyses, and other events that do not fall within the standard operating procedures. A “Memo-to-file” will be used to document any decisions or corrections that are made. This memo will include the date, name of author, site or sample referred to, a description of the problem or error and a statement describing the decision made or action taken. The memo will be archived with the appropriate site data and files.

12.13 Reports to Management

Routine data summaries will occur annually for lakes. Summary reports will be produced periodically. Web-based visualization and data summary tools for inland lake water quality data are under development. These tools and summary reports will be provided to park resource managers, park partners, and the public.

More comprehensive analyses of trends will occur for most parameters after three or more years of sampling. For stations that are located where no previous monitoring has occurred, this period of time tends to be the minimum needed to establish a time series sufficiently powerful to detect meaningful levels of change (e.g., 20%) through time. The target audience of the analysis and synthesis reports will be the parks, the network, both regional and Service-wide I&M, and the broader scientific community. Drafts will be reviewed internally and sent to the parks, and possibly

outside sources, for further review. The extent of review will depend on how analytically complicated the methods are and the gravity of inference and recommendations.

12.14 Data Validation, Certification, and Usability

Data validation is the method in which data are proven or disproved to be accurate. This process involves the review of the results of all measurements, samples, and QC samples. Field data sheets and laboratory data are reviewed for transcription errors, completeness, verification of calibration and quality control check samples or standards.

A checklist for use in data validation is presented in Attachment B. Data are validated by comparing the actual estimates for MDL and ML, precision, bias/uncertainty for each analytical run to the expected quality of results. Once this information is documented, the decision is made by the project manager on the usability of the data. Data may be flagged as between MDL and ML and therefore highly uncertain (J). Data may be flagged (B) if the analyte was detected in the blank at a concentration similar to that in the sample (laboratory flagging rules may differ slightly). These types of flagged data contain much more uncertainty than unflagged data. The decision must be made whether or not to add them to the database or merely maintain them in files for future reference. Some investigation into the sampling method or laboratory method may be needed if ‘B’ flagged data are a continual occurrence or if the required MLs are repeatedly unmet.

NPSTORET itself includes some form-based QA/QC tests on data entry. The system does not allow entry of results below the ML but will flag the data as “present, not quantified”. The project manager, in consultation with other NPS or outside experts, will flag data when they fall outside of expected limits. Laboratory results below detection limits (ND) will be kept in the archives as ‘ND’ and will be handled statistically according to recent literature (e.g., Helsel 2005) in summary and analysis reports. Aquarius provides QA/QC tools to check for single value or rate of change data outliers, for example.

Once returned to GLKN by WRD, and after appropriate documentation is completed, the NPSTORET datasets are officially considered certified. A process for certifying data imported into Aquarius is being developed by WRD. Data certification is a benchmark in the project information management process that indicates that: 1) the data are complete for the period of record; 2) they have undergone and passed the quality assurance checks; and 3) that they are appropriately documented and in a condition for archiving, posting and distribution as appropriate.

Certification is not intended to imply that the data are completely free of errors or inconsistencies, which may or may not have been detected during quality assurance reviews. Only qualified users who have been trained and given edit permissions are allowed to edit data in NPSTORET or Aquarius. These procedures protect the integrity of the data and allow the history of each data record to be traced.

12.15 Reconciliation with Data Quality Objectives

Once data validation is complete, the project manager then calculates percent completeness, compares the results to the data quality objectives, and determines whether or not the data meet the objectives for both the field and laboratory components of the project.

12.16 Literature Cited

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Attachment A. Checklists for Selection of Multiprobe and Selection of Laboratory.

Multiprobe Checklist

Probe _____ Purchased _____

Serial Number _____ Location used _____

Manufacturer's Stated Limits

Sensor	Range	Reporting Resolution	Bias	Stabilization Criteria
Temperature				
Sp. Cond. (SC25)				
pH				
DO (Conc.)				
DO (% sat.)				
Depth – Z (pressure sensor)				

Alternative measurement sensitivity by multiple field readings

Location _____ Performed by _____

Date _____ Began calibration log _____

Sensor	1	2	3	4	5	6	7
Temperature							
Specific Conductivity (SC25)							
pH (sensor units)							
DO (Conc.)							
DO (% sat.)							
Depth – Z (pressure sensor)							

Calculations

For each parameter calculate the standard deviation of the 7 readings.

Sensor	Standard deviation	AMS 3.708x sd		
Temperature				
Specific Conductivity (SC25)				
pH (sensor units)				
DO (Conc.)				
DO (% sat.)				
Depth – Z (pressure sensor)				

Alternate Measurement Sensitivity is a two-sided estimation based on the 99% confidence interval for sample size 7. Of interest is the size of the difference between two individual values that can be considered to actually be a true difference. The MDL is a one-sided true difference from zero.

Contract Laboratory Checklist

Laboratory:

Address:

Contact person:

_____ Received QAPP

_____ Received copy of certifications

_____ Received a list of analytical methods used

_____ Define the limits of detection and limit of quantitation calculation method
(can lab report as MDL and ML if currently calculated as LOD and LOQ?)

_____ Received a copy of latest MDL/ML/control limits calculations for relevant methods

_____ MDL/ML as listed meet project needs
(list any analyte for which the ML requirement is not met – discuss options with lab)

_____ Received successful interlaboratory participation documentation

_____ Sample handling log in and COC are documented in the QAPP

_____ Equipment maintenance and calibration procedures are documented in the QAPP

_____ Internal QA/QC documented in QAPP

Control limits calculations are made by what method
(QC includes blanks, duplicates, spikes, reference standards and LCS)

Calibration curves cover level of analytical interest
(QC includes ICV and ICB, and CCV and CCB)

Reporting data flags used include

Attach all copies

Reviewed by _____

Date _____

Attachment B. Checklists for data validation.

Field Measurement Validation Checklist

(one sheet per Park sampled)

Sampling Unit _____

Date Sampled _____

Date Reviewed _____

Reviewed by _____

_____ All field forms have been received (data sheet, flow, etc.)

_____ Multiprobe was calibrated correctly

_____ Multiprobe post-calibration checks were successful

_____ Field duplicates were within range

_____ Multiprobe end of use calibration checks were successful

_____ There were no obvious trends in data taken from any sensor during the sampling day

_____ Equipment blanks sent to lab included

Laboratory Data Validation Checklist

Sample set from _____

Date taken _____

Received data _____

Reviewed by _____

Review date _____

_____ Samples received by lab at proper temperature (look at COC copy from lab)

_____ Holding time limits met

_____ Analytical methods used in analyses were those agreed upon

_____ Useable MDL and ML achieved in this analytical run

_____ Calibration procedures were followed

QC samples control limits applicable to this analytical batch

Lab blank _____

Lab dup _____

Lab LCS _____

Lab spikes _____

Field dup _____

Eq blank _____

CCVs _____

QC samples within range expected

Lab blank _____

Lab dup _____

Lab LCS _____

Lab spikes _____

Field dup _____

Eq blank _____

CCVs _____

Lab notes or flags

Samples rejected

Signature of Reviewer _____