



Quality Assurance Plan for Monitoring Mercury in Dragonfly Larvae and Fish

Natural Resource Report NPS/GLKN/NRR—2018/1697

DRAGONFLY Field Sheet

National Park name: APIS Site Location: _____
Site name/Waterbody: Stockton Island Lagoon Site Location: _____
Date: 9/15/2016 Lead collector name/Contact info: RANDY LEHR

Dragonfly Samples collected:			Size (mm)
#	Sample ID	Family	
1	16APIS0016	Aeshnidae	22 mm
2	16APIS0017	Gomphidae	20 mm
3	16APIS0018	Aeshnidae	20 mm
4	16APIS0019	Gomphidae	29 mm
5	16APIS0020		
6	16APIS0021		
7	16APIS0022		
8	16APIS0023		
9	16APIS0024		
10	16APIS0025		
11	16APIS0026		

Sample: 16APIS0021
Date: 9/15/16
Dragonfly length: 19 mm

Dragonfly family:
☐ Libellulidae
☒ Corduliidae
☐ Gomphidae
☐ Aeshnidae
☐ Macromiidae
☐ Cordulegasteridae
☐ Unknown

Naturalist: _____
Notes: _____

ON THE COVER

Field staff transcribe information from the data sheet to a tag that will accompany the dragonfly larvae.

NPS photo / T. Gostomski

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August 2018

U.S. Department of the Interior
National Park Service
Natural Resource Stewardship and Science
Fort Collins, Colorado

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Please cite this publication as:

VanderMeulen, D. D., S. J. Nelson, A. Klemmer, C. Eagles-Smith, and J. Willacker. 2018. Quality assurance plan for monitoring mercury in dragonfly larvae and fish. Natural Resource Report NPS/GLKN/NRR—2018/1697. National Park Service, Fort Collins, Colorado.

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Abstract

In accordance with guidelines set forth by the National Park Service Inventory and Monitoring Division, a quality assurance plan has been created for use by the Great Lakes Inventory and Monitoring Network (GLKN) in the implementation of monitoring mercury in dragonfly larvae and fish. This quality assurance plan documents the standards, policies, and procedures used by the GLKN for activities related to the collection, processing, storage, analysis, and publication of monitoring data associated with monitoring mercury in dragonfly larvae and fish. The policies and procedures documented herein complement the quality assurance plans and procedures for other monitoring activities conducted by the GLKN and supplement the National Inventory and Monitoring Division Quality Management Plan.

List of Terms and Abbreviations

Terms

Accepted Data. Data that have undergone all QA and QC procedures as defined in a published protocol and associated SOPs. Used in cases where a Quality Assurance Plan has not been developed and approved.

Accreditation. A formal recognition that an individual or organization (e.g., laboratory) is competent to carry out specific tasks or specific types of tests, and/or meets predetermined qualifications or standards. For labs, this is typically done on an analyte basis for a limited/specific period of time.

Accuracy. A measure of the degree of conformance of the values generated by a specific method or procedure with the true value. The concept of accuracy includes both bias (systematic error) and precision (random error). EPA recommends that this term not be used and that precision and bias be used to convey the information usually associated with accuracy. See Precision and Bias.

Bias. Systematic error that is manifested as a consistent positive or negative deviation from the known or true value. It differs from random error which shows no such deviation.

Calibration. Determination, by measurement or comparison with a standard, the correct value of each measure on a meter or other device, or the correct value for each setting of a control knob. The levels of the calibration standards should bracket the range of planned measurements.

Certified Data. Certified data are those data that a) have been through all QA/QC procedures as outlined in the QAP, b) meet expected quality standards, or are appropriately identified and annotated at the record level in cases where they do not meet those standards, c) have documented procedures for data collection, processing, and transformation in appropriate metadata formats, and d) are suitable for use.

Comparability. The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator.

Data Quality Objectives (DQOs). Qualitative and quantitative statements that clarify the objectives of observations, define the appropriate type of data, and specify levels of uncertainty that are acceptable to managers when using data. DQOs provide the statistical framework for planning data collection and management, and are used as the basis for establishing the quality and quantity of data needed to support decisions. Includes Qualitative Data Quality Values (DQVs), and informs development of Measurement Standards (or Measurement Quality Objectives; MQOs), Performance Standards, Accreditation Standards, and Policy Standards.

Data Quality Values (DQVs). Desired qualitative characteristics of data and datasets related to intrinsic data quality (reasonability, accuracy, objectivity, credibility), contextual data quality (relevance, timeliness, completeness, comparability), representational data quality (interpretability, understandability, consistency), and data accessibility (security, discoverability, utility).

Data Validation. Quality Control tools or processes designed to evaluate legitimacy of data as they proceed through the data management lifecycle. Typically data that do not pass validation procedures are either corrected using prescribed methods or flagged/graded to inform data users as to their appropriate use.

Data Verification. Quality Assurance tools or processes where data are checked for accuracy and inconsistencies as they migrate through the data management lifecycle. Data Verification helps to ensure that data are complete, properly formatted so that they can be processed, and accurately translated when transferred from one source to another.

Data Flagging / Data Qualification. A code or annotation that signals that data do not meet specified data quality standards so as to inform appropriate use.

Minimum Detection Limit (MDL). The minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, qualitatively or quantitatively measured, and reported to be greater than zero.

Minimum Quantification Limit (MQL). The concentration of analyte in a specific matrix for which the probability of producing analytical values above the method detection limit is 99 percent.

Precision. The degree of similarity among independent measurements of the same quantity, without reference to the known or true value.

Provisional Data. Provisional data are those data that have undergone incomplete QA/QC procedures as defined in the QAP and are not suitable for general use. Provisional data may be used in some cases, however, depending on the amount of QA/QC completed, and the intended use.

Quality Assurance (QA). The set of activities designed to ensure that data, products, or services meet defined standards of quality with a stated level of confidence. Quality Assurance focuses on ensuring that data are collected to and managed to the highest practicable quality, at or above stated data quality standards.

Quality Assurance Plan (QAP). Documentation of Quality Assurance and Quality Control procedures for a protocol, program (inventory and overall I&M), and national database application (i.e., Data Store).

Quality Control (QC). Operational techniques and activities that are used to ensure that data or products meet designated requirements for quality. Quality Control procedures focus on

testing data for quality and can include both an evaluation of whether data quality meet data quality objectives and/or procedures to correct data.

Raw Data. Data in their state of original collection, either in the form of paper or electronic field forms, electronic sensor files, or digital media (photo, video, or audio). These data generally have been collected following some QA procedures, but have not undergone any QC procedures.

Sensitivity. The ability of a method or instrument to discriminate between minimally different levels of a variable of interest by producing a noticeably different measurement response.

Standards, Measurement (also Measurement Quality Objectives). Data quality requirements specifying the level of precision, bias, sensitivity, resolution, etc. that data must meet to be accepted for use in analysis.

Standards, Performance. Data quality requirements specifying the detection ability, sensitivity, variability, etc., of equipment, sensors, or observers to reliably measure characteristics within target populations.

Standards, Accreditation. Data quality requirements related to individuals or organizations involved with data collection, analysis or processing to ensure desired data credibility and defensibility. Examples include professional accreditation/certificates, required training, chain-of-custody requirements and others.

Standards, Policy. Data quality requirements related to ensuring appropriate management, use, dissemination, qualification, and documentation of data.

Abbreviations

National Park Service

APIS	Apostle Islands National Lakeshore
GLKN	Great Lakes Inventory and Monitoring Network
GRPO	Grand Portage National Monument
INDU	Indiana Dunes National Lakeshore
ISRO	Isle Royale National Park
MISS	Mississippi National River and Recreation Area
NPS	National Park Service
NPSTORET	National Park Service Storage and Retrieval Database
PIRO	Pictured Rocks National Lakeshore
SACN	St. Croix National Scenic Riverway

SLBE	Sleeping Bear Dunes National Lakeshore
VOYA	Voyageurs National Park
WRD	Water Resources Division of the National Park Service
<i>Additional Abbreviations</i>	
CCV	Continuing Calibration Verification
DQO	Data Quality Objective
DQV	Data Quality Value
m	Meter
MDL	Minimum Detection Limit
MeHg	Methylmercury
MQL	Minimum Quantitation Limit
MQO	Measurement Quality Objective
ng/g	Nanograms per gram
PD	Percent Difference
pg	Picograms
QA/QC	Quality Assurance/Quality Control
QAP	Quality Assurance Plan
Reference standard	Independently created solution of known concentration.
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
STORET	Storage and Retrieval Database
THg	Total mercury
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey

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 quality assurance plan 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 Procedure for Revising the Protocol, SOP #9 in the protocol.

Revision History Log:

Previous Version #	Revision Date	Author (with title and affiliation)	Location in Document and Concise Description of Revision	Reason for Change	New Version #
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
Add rows as needed for each change or set of changes tied to an updated version number					

Acknowledgments

Most of the organizational structure of this quality assurance plan is derived from a draft template provided by J. DeVivo, Deputy Chief for Science, Inventory and Monitoring Division. Many helpful comments were provided by K. Bartz, Aquatic Ecologist, Southwest Alaska Inventory and Monitoring Network.

Introduction

The National Park Service (NPS) Inventory and Monitoring Program was established by an act of Congress in 1999 to provide more than 270 national park system units a core suite of baseline inventories and to identify and implement long-term monitoring of high-priority natural resources (Fancy et al. 2009). The overarching purpose of natural resource monitoring in parks is to develop scientifically sound information on the current status and long-term trends in the composition, structure, and function of park ecosystems, and determine how well current management practices are sustaining those ecosystems.

In accordance with guidelines set forth by the National Park Service Inventory and Monitoring Division, this quality assurance plan (QAP) was developed by the Great Lakes Inventory and Monitoring Network (GLKN) to accompany a protocol for monitoring mercury in dragonfly larvae and fish (version 1.0; hereafter referred to as “the protocol”). The purpose of this QAP is to document the standards, policies, and procedures used by the GLKN for the collection, processing, storage, analysis, and publication of data under the protocol. This QAP serves as a guide for all personnel who are involved in field, lab, and office activities and as a resource for identifying memoranda, publications, and other literature that describe associated techniques and requirements in more detail. The policies, procedures, and activities documented herein complement the quality assurance plans for other monitoring activities conducted by the GLKN and supplement the National Inventory and Monitoring Division Quality Management Plan currently in development.

Protocol Overview

The protocol for monitoring mercury in dragonfly larvae and fish (version 1.0) involves collection of these biosentinel organism and analyzing either the whole organism (larval dragonflies and prey fish) or axial muscle tissue (skinless filet of predatory fish) for mercury. Mercury is also a metal that when methylated biomagnifies to potentially toxic concentrations in upper trophic levels. Atmospheric transport and deposition are the primary pathways for entry of mercury into most watersheds and surface waters in the western Great Lakes region (e.g., Wiener et al. 2006). Although most of the mercury in atmospheric deposition exists as inorganic forms, nearly all of the mercury accumulated by larval dragonflies, fish, and other top predators is methylmercury (Grieb et al. 1990, Bloom 1992, Hammerschmidt et al. 1999, Van Walleggem et al. 2007, Nelson et al. 2015, Wiener et al. 2016). Methylmercury is an organic compound produced by anaerobic bacteria that are present in wetlands, sediment, and anoxic bottom waters (Benoit et al. 2003, Colombo et al. 2013, Gilmour et al. 2013, Parks et al. 2013). Mercury levels in biosentinel organisms vary greatly among aquatic ecosystems, including those in close proximity, due to a number of factors that affect mercury methylation and delivery to the base of the aquatic food web and that affect biomagnification (e.g., Brigham et al. 2014). Both larval dragonflies and fish are high on the aquatic food web and bioaccumulate mercury. Hence they sample the aquatic environment and act as integrators of mercury contamination.

The GLKN first began monitoring mercury in bald eagles, another biosentinel organism, at Apostle Islands National Lakeshore (APIS), Mississippi National River and Recreational Area (MISS), and St. Croix National Scenic Riverway (SACN) in 2006, with annual sampling occurring through 2011,

and again in 2014 and 2015 (W. Route pers. comm.). From 2008 to 2012 larval dragonflies and fish were collected from Grand Portage National Monument (GRPO), Indiana Dunes National Lakeshore (INDU), Isle Royale National Park (ISRO), Pictured Rocks National Lakeshore (PIRO), Sleeping Bear Dunes National Lakeshore (SLBE), and Voyageurs National Park (VOYA), and analyzed for mercury (Wiener et al. 2016).

Separately, in 2011 the NPS Air Resources Division partnered with the University of Maine and 4 national parks across the U.S. and began a citizen-science based pilot project to collect and analyze larval dragonflies for mercury. This work was expanded to 11 parks in 2012. One of the objectives of the project was to evaluate relationships among potential factors that influence mercury accumulation in larval dragonflies and mercury in water across the broad spatial scale of parks in all NPS regions to determine their utility as biosentinels of exposure (Nelson et al. 2015). Since 2012, 93 parks, including all nine of the GLKN parks, have participated in this project (C. Flanagan-Pritz pers. comm.). Through related work, in 2015 and 2016, fish were also collected from a number of parks in the eastern U.S., including INDU, ISRO, MISS, SACN, and VOYA, and are being analyzed for mercury (Collin Eagles-Smith, USGS, unpublished data).

Prior results of this work have been published in technical reports (Nelson et al. 2015, Wiener et al. 2016) and science publications (Haro et al. 2013, Brigham et al. 2014, Rolfhus et al. 2015, Sandheinrich and Drevnick 2016). Through the protocol, we will build on these earlier efforts and monitor mercury levels in dragonfly larvae annually and prey and/or predatory fish every five years at all nine GLKN parks.

The monitoring program is designed to answer the following questions:

- 1) Which park units and surface waters within the GLKN have concentrations of mercury in dragonfly larvae and fish high enough to pose a risk to humans and wildlife in upper trophic levels?
- 2) What are the spatial patterns in mercury contamination in dragonfly larvae and fish within parks of the GLKN?
- 3) What is the general direction and magnitude of change in the concentration of mercury in dragonfly larvae and fish in parks of the GLKN?

Briefly, 20 larval dragonflies per site are sampled from up to four aquatic sites (three fixed and one rotating) per park annually in the summer. Rationale for the minimum number of larval dragonflies and fish (below) is described in Appendix B of the protocol. The site locations within each park unit incorporated substantial input from park personnel as well as the following general criteria: (1) to select study sites with available historical mercury data and pertinent ancillary information on the biological, physical, and chemical characteristics of aquatic resources, (2) to maximize spatial overlap between mercury monitoring and other aquatic monitoring by GLKN, and (3) to reduce logistical obstacles to sampling. Information such as pH, temperature, dissolved oxygen, water clarity (measured with Secchi disk, transparency tube, or levels of total suspended solids), major ions, nitrogen, phosphorus, bathymetry, water level, hydrology, and landscape variables (drainage

area, forest type, and wetlands area) for lakes or rivers routinely monitored for water quality by GLKN can be found in Elias et al. 2015 or Magdalene et al. 2016.

To maximize efficiency, many parks will collect larval dragonflies during previously scheduled GLKN water quality monitoring, typically June through August. This schedule is consistent with park and citizen science-based larval dragonfly monitoring (Nelson et al. 2015, Eagles-Smith et al. 2016) that has taken place annually at GLKN parks since 2012. Larger (length ≥ 15 mm) larvae are targeted, as they are more likely to integrate mercury conditions during the previous two to five years, depending on their taxonomy. The field crew uses dip-nets to collect larvae from bottom substrates, overhanging banks, and aquatic vegetation in the littoral zone of lakes, rivers, and streams. Once collected, larvae are identified to family, measured (length to nearest mm), placed in individually labeled and sealed plastic bags, and kept on wet ice until they are frozen. Samples are then sent to an analytical laboratory where taxonomy to family and length are verified, wet and dry weights obtained, and total mercury concentrations determined.

Fish are sampled every five years at each park, on a rotation of two GLKN parks per year, except for the fifth year when only one park is sampled. Sampling sites overlap with those sampled for larval dragonflies. Collection methods used (e.g., electrofishing, angling, seining, etc.) depend on the type of fish (i.e., prey or predatory fish). Fish sampling likely will not overlap with other monitoring activities as it is time and labor intensive. Prey fish ($n \geq 20$ per site) will be collected in the spring a few weeks after ice out, when growth is slow and temporal variation in mercury concentration is small, to obtain data that are temporally and spatially comparable. Predatory fish ($n \geq 15$ per site) will be collected throughout the summer. Like dragonfly larvae, fish samples are also collected and examined as individuals. They are identified to species, kept on wet ice until frozen, and sent to an analytical laboratory to be measured, weighed, and assessed for total mercury. All data for larval dragonflies and fish are recorded on standardized forms.

Resulting data are validated, assessed for quality, certified, and imported into a Microsoft Access database called NPSTORET. This database is used to store, document, and transfer data. The GLKN oversees the use of NPSTORET per the GLKN's aquatic monitoring protocols and ensures that data are transferred at least annually to the NPS WRD for upload to the EPA STORET Data Warehouse and the National Water Quality Monitoring Council's (NWQMC) Water Quality Portal.

Summary of Data Collection Activities

Data are collected under this protocol during the following seven activities:

- 1) Site Characteristics. In addition to collection of biological samples, GLKN or park field crews document site characteristics for each monitoring location at the onset of each sampling event. Along with standard information such as names of field crew members, date, time, and GPS coordinates, site information such as surface water type, category of substrate in the littoral zone, surrounding land use, percent canopy cover, etc. are also recorded. Site characteristics are part of the metadata record for each sampling event and help document how gradual (e.g., infilling of a shallow pond by emergent aquatic vegetation) or abrupt (e.g.,

beaver dam blowout leading to lower water levels) changes may be impacting dragonfly or fish communities.

- 2) Biological Samples - Larval Dragonflies. At least 20 larval dragonflies are collected from each aquatic site by GLKN or park staff. The larvae are identified to family, measured (length to nearest mm), placed in individually labeled and sealed plastic bags, and kept on wet ice until they are frozen. The field crews follow detailed standard operating procedures for collection (SOP #4, Fieldwork – Dragonfly Larvae and Fish), with sample information for individual larvae recorded on a standardized form. Samples are shipped frozen to an analytical laboratory.
- 3) Biological Samples - Fish. At least 20 prey or 15 predatory fish are collected from each aquatic site by GLKN or park staff. Fish are identified to species, measured (length to nearest mm), placed in individually labeled and sealed plastic bags, and kept on wet ice until they are frozen. The field crews follow detailed standard operating procedures for collection (SOP #4, Fieldwork – Larval Dragonflies and Fish), with sample information for individual larvae recorded on a standardized form. Samples are shipped frozen to an analytical laboratory.
- 4) Laboratory Measurements - Larval Dragonflies. At an analytical laboratory dragonfly larvae are thawed on a clean, acid-washed work surface and measured (total length to the nearest mm); those with body lengths ≥ 15 mm are processed and analyzed individually. Smaller individuals of a given species with body lengths < 15 mm can be pooled into composite samples for analysis at the analytical laboratory's discretion, given instrument technical specifications. Individual larvae are rinsed briefly in deionized water to remove surficial debris then blotted with a lint-free wipe to remove excess surface moisture prior to obtaining a wet weight (ww) (to 0.001 g). Dragonflies can be freeze-dried or dried in a laboratory oven set to 50°C. Once a constant mass is obtained (typically 48 hours) a final dry weight (dw) (to 0.001 g) is recorded. Depending on the size of individual larvae, samples are analyzed for total mercury either whole or ground using either acid-washed mortar and pestles or glass rods.
- 5) Laboratory Measurements - Prey Fish. At an analytical laboratory prey fish are gradually thawed at room temperature, identified to species, measured (total length to the nearest millimeter), and weighed (to 0.001 g). Samples are then coarsely chopped to facilitate rapid and complete moisture loss, and dried in a laboratory oven set to 50°C or in a freeze dryer. Once a constant mass is obtained (typically 48 hours) a final dry weight (dw) (to 0.001 g) is recorded. After drying, double-bagged samples are stored in a desiccator with sufficient drying agent and vacuum. Each dry sample should be homogenized to a fine powder using an acid mortar and pestle and analyzed for total mercury.
- 6) Laboratory Measurements – Predatory Fish. At an analytical laboratory predatory fish are identified to species, weighed (to 0.001 g), measured (maximum total length), and dissected to remove an approximately 5 g sample of axial muscle tissue. Samples of axial muscle will be stored frozen until drying as described above for whole prey fish. Once the tissue is

lyophilized a final dry weight (dw) (to 0.001 g) is recorded. Dried muscle samples are homogenized to a fine powder using an acid washed mortar and pestle and analyzed for total mercury.

- 7) Derivative and Miscellaneous Data. Some values are derived from the raw data. For example, descriptive statistics for total mercury concentration should include geometric mean, median, maximum and minimum values, skewness, kurtosis, and measures of variability (e.g., coefficient of variation, standard error, or variance).
- 8) QA/QC data. GLKN and park staff follow QA/QC procedures outlined in this document as well as the SOPs associated with the monitoring protocol. The analytical laboratories are required to have an approved laboratory QAP, and provide QA/QC data on results for every batch of samples.

Data Quality Objectives

Data Quality Values

Data quality values and standards for implementation are provided in Table 1; measurement quality objectives are provided in subsequent sections.

Table 1. Data Quality Values (DQVs) for dragonfly larvae and fish measuring and sampling.

Data Quality Type	Data Quality Value	Definition	Protocol Considerations
Intrinsic Data Quality	Accuracy	Measurements reflect the true value of the parameter being observed. This applies to measures (length, width, position) or classes (species, types, or categories). Includes components of precision and bias.	Data collected in the field are assumed to be accurate representations of the true values. Repeated sampling provides reasonable estimates of the true variability of mercury in larval dragonfly and fish. Laboratories provide measures of accuracy as part of method development and quality control.
	Representativeness	Measurements represent conditions at the time of sampling. Combined with accuracy, leads to repeatable data collection.	Methodological considerations such as timing of sampling, sampling gear, and sampling handling are taken into account to ensure data are representative in any given year.
Contextual Data Quality	Comparability	The degree to which data can be compared among sample locations, data sources, or periods of time.	Nearly all the sites have been previously monitored using identical methods, which provides an excellent baseline for future comparisons.
	Timeliness / Currency	How recent the data need to be to be considered valid for their intended use. Data represents conditions and/or is available and in a format for use at the appropriate time in the decision-making process.	Laboratory results can take up to a year to obtain and analyze. Preliminary results are provided on request or if there is a risk to human health. Final mercury results can take up to three years before publication.
	Completeness	All data/ measures required to evaluate accuracy representativeness are present; incomplete data sets (either at a location, across sampling locations, or over time) lose utility or relevance. Data records contain values as planned across the period of record.	The protocol is designed to provide complete data sets across space and time. However, inability to collect enough specimens, laboratory equipment problems, or some other unforeseen loss of samples (e.g., an undelivered cooler) may result in missing values. Data analysis procedures are available to handle missing values.

Table 1 (continued). Data Quality Values (DQVs) for dragonfly larvae and fish measuring and sampling.

Data Quality Type	Data Quality Value	Definition	Protocol Considerations
Representational Data Quality	Consistent Representation	Use of standard definitions when describing data quality or resource quality based on data	Provisional data are data that have been reviewed but not certified and are used only for preliminary evaluations or analysis steps towards certification. Certified data have gone through all QA/QC steps and can be used for analysis and reporting. For laboratory data, certification includes a Quality Assurance Report (described below). Standard definitions as shown in this document will be used.
Data Accessibility	Secure	Access to data, products, and systems limited to appropriate audiences.	Once certified, all data collected through this protocol will be available to the public through the U.S. EPA's Water Quality Exchange. The NPS Water Resources Division and GLKN utilize NPSTORET, a Microsoft Access database tool, which duplicates most of the U.S. EPA data and table structures to facilitate easier movement of GLKN's mercury data into the EPA Water Quality Exchange framework.

Measurement Quality Objectives - Fieldwork

Measurement Quality Objectives for fieldwork associated with sampling dragonfly larvae and fish are shown in Table 2 and Table 3, respectively.

Table 2. Measurement Quality Objectives for fieldwork associated with collection of larval dragonflies.

Category	Measure / Quality Indicator	Quality Objective
Timing of Sampling	Annually, May-October	Larval dragonflies are sampled annually, typically June through August, and in some cases coincident with other monitoring efforts. If necessary they can be sampled earlier in the spring or later in the fall. However, all sampling within a given calendar year should occur sometime within the May through October time period.
Site Locations	Location Accuracy	The locations –including selection rationale–, of all fixed aquatic monitoring sites (three per park) are provided in the protocol narrative. One site is allowed to rotate from one year to the next, at the parks' discretion. Previously-established GPS locations for these sites are used to navigate to sampling locations from prior years, with the assumption that mercury concentrations in larval dragonflies collected from one area within an aquatic site are representative of mercury concentrations in larval dragonflies site-wide.
Site Characteristics	Date/time of sample collection, field crew names, surface water type, category of substrate, surrounding land use, and percent canopy cover	Site characteristics are recorded during each site visit and included as part of the metadata associated with sample results. Data such as date and time of sampling and names of field crew members are recorded; other data such as surface water type, surrounding land use, and percent canopy cover are subjective and selected from a descriptive list. Any other conditions that have the potential to affect mercury levels at the site (beaver dam blowout, gradual infilling of a shallow pond with emergent vegetation, etc.) are also noted. For each sampling effort all fields related to site conditions are completed.
Sample Size	Number of larval dragonflies per site	Following detailed collection procedures outlined in SOP #4, Fieldwork – Dragonfly Larvae and Fish, the objective is to collect at least 20 larval dragonflies at each aquatic site.
Individual Measurements	Length	The total length of each individual larval dragonfly is measured to the nearest mm using a 35-cm ruler. Data are recorded on field sheets. Comparisons between observers in the field suggest these measurements can vary by up to 1 mm.

Table 2 (continued). Measurement Quality Objectives for fieldwork associated with collection of larval dragonflies.

Category	Measure / Quality Indicator	Quality Objective
Sample Identification	Sample ID	Each individual larval dragonfly is assigned a sample ID using the following convention: GLKN_4-letter park acronym_2-digit site code_3-digit sample number. Use the site codes previously assigned for GLKN water quality monitoring (inland lakes and large rivers) as shown in NPSTORET. For example, the eleventh larval dragonfly collected from Lake Richie (03) in Isle Royale National Park (ISRO) would be labeled "GLKN_ISRO_03_011".
	Sample Taxonomy	Field sampling crews will identify individual dragonfly larvae to family and record on the field sheet. There are only six major families in GLKN parks (see Attachment B in SOP #4, Fieldwork – Dragonfly Larvae and Fish). Most identifications can be accomplished in the field with a hand lens and guide book. A link to an online key is available at http://nature.nps.gov/air/Studies/air_toxics/dragonfly/ will assist with identification efforts. If necessary, samples can be identified in a clean indoor workspace prior to freezing them for shipment.

Table 3. Measurement Quality Objectives for fieldwork associated with collection of fish.

Category	Measure / Quality Indicator	Quality Objective
Timing of Sampling	Annually, May-October	Fish are sampled at each GLKN park every five years. During a sampling year fish are typically collected June through August, and in some cases coincident with other monitoring efforts. If necessary fish can be sampled earlier in the spring or later in the fall. However, all sampling within a given calendar year should occur sometime within the May through October time period.
Site Locations	Location Accuracy	Fish sampling locations are coincident with the three fixed aquatic sites where dragonflies are monitored annually. The location of the fourth site is at the parks' discretion; it most likely coincident with whatever parks' choose to be their rotating site for larval dragonflies in the year in which fish are also sampled. Previously-established GPS locations for these sites are used to navigate to sampling locations from prior years, with the assumption that mercury concentrations in fish collected from one area within an aquatic site are representative of mercury concentrations in fish site- (i.e., lake)-wide.

Table 3 (continued). Measurement Quality Objectives for fieldwork associated with collection of fish.

Category	Measure / Quality Indicator	Quality Objective
Site Characteristics	Date/time of sample collection, field crew names, surface water type, category of substrate, surrounding land use, and percent canopy cover	Site characteristics are recorded during each site visit and are included as part of the metadata associated with sample results. Data such as date and time of sampling and names of field crew members are also recorded; other data such as surface water type, surrounding land use, and percent canopy cover are subjective and are selected from a descriptive list. Any other conditions (beaver dam blowout, gradual infilling of a shallow pond with emergent vegetation, etc.) are also noted. For each sampling effort all fields related to site conditions are completed.
Sample Size	Number of fish per site	Following detailed collection procedures outlined in SOP #4, Fieldwork – Dragonfly Larvae and Fish, the objective is to collect at least 15 predatory fish (typically Northern Pike) or 20 prey fish (typically Yellow Perch) at each aquatic site.
Individual Measurements	Length	The total length of each individual fish is measured to the nearest mm using a ruler. Data are recorded on field sheets. In some instances where typical predatory fish (Northern Pike, Smallmouth Bass) are absent (e.g., Lake Harvey at ISRO), large Yellow Perch are considered as the top predatory fish as long as they are ≥ 100 mm in length.
Sample Identification	Sample ID	Each individual fish will be assigned a sample ID using the following convention: GLKN_4-letter park acronym_2-digit site code_4-letter fish acronym assigned by the American Fisheries Society_3-digit sample number. Use the site codes previously assigned for GLKN water quality monitoring (inland lakes and large rivers) as shown in NPSTORET. For example, the ninth Northern Pike collected from Lake Richie (03) in Isle Royale National Park (ISRO) would be labeled “GLKN_ISRO_03_NOPI_009”.
	Sample Taxonomy	Field sampling crews will identify individual fish to species. Fish species, -especially since in nearly all instances we are targeting a single species (for predatory fish, Northern Pike, or prey fish, Yellow Perch)-, are easily identified.

Preventing Sample Contamination

It is easy to contaminate samples with mercury because it is all around us: in our hair, in soil, leaves, etc. We need to keep everything ultra-clean in order to avoid contamination of samples and biasing sample results. The following procedure should be used when handling biological samples:

- 1) Do not touch the samples with anything except a clean plastic spoon (individual dragonfly larvae) or powder-free nitrile gloves (all samples).

- 2) Never touch the inside of the inner bag – the first bag you put the sample into.
- 3) Think of each individual sample as a fresh start – everything needs to be clean between samples and be treated just like the first sample you took.
 - a) Work in pairs and use a “Clean Hands–Dirty Hands” method:
 - b) One person is in charge of the sample bags (“Dirty Hands”) and the other will pick up and place the samples in the bags (“Clean Hands”).
 - c) Clean Hands puts on a glove (one gloved hand should suffice to pick up and bag sample). Once the glove is on, Clean Hands touches nothing but the sample and any pre-cleaned supplies. Dirty Hands does not need to wear gloves.
 - d) Choose, measure, and bag the requisite number of samples per waterbody/site.
 - e) Using a nitrile-gloved hand, (or plastic spoon), Clean Hands places one sample into a sample bag while Dirty Hands holds the bag. Nothing should touch the inside of the bag except the sample. Clean Hands seals the bag.
 - f) Dirty Hands measures the body length (to the nearest mm).
 - g) Dirty Hands fills out a label for the bagged individual and slips it and the inner bag into the outer bag. Use the same sample ID that is on the label to record the lengths on the field sheet and to refer to a particular sample in your notes.
 - h) Dirty Hands seals the outer bag.
 - i) Be ‘clean’ with your spoon and/or gloves. If they have been tossed on the ground or contaminated – say, by brushing back your hair – start with a fresh one. Swish your spoon or gloved hands in water from the site (in the dishpan) between samples to ensure there’s no carryover from one sample to the next.
 - j) Go to the next individual and repeat. Place all of the small double bags into one or more of the larger zipper seal bags for storage and to keep them safe and together.
 - k) Gently return extra site water and extra samples to the sample site.

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.

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

Completeness will also be calculated annually for number of samples collected compared to those that have been analyzed for mercury and recorded.

Measurement Quality Objectives – Laboratory

Field equipment and contract laboratories are likely to change during the course of this long-term monitoring project. Documentation of the contract laboratory's ability to meet the data quality objectives of this project will be maintained (Attachment A). Documentation including each laboratory's QAP will be maintained in project files for each analytical laboratory.

Physical Measurements

Upon receipt of dragonfly larvae, prey fish, or adult predatory fish, the analytical lab will allow the samples to thaw and if necessary remove any moisture by patting dry with a lint-free wipe. Each individual sample is measured for total length (to the nearest mm) and weighed to determine wet (fresh) weight (to 0.001 grams). Measured lengths will be checked against reported length by field crews on the field forms and sample labels. In some instances the lab may sex the larval dragonflies, by examining secondary sexual characteristics, including swellings on abdominal segments 2 and 3 in male larvae (Needham et al. 2000). An axial muscle tissue (skinless fillet) sample will be removed from each predatory fish. All samples (dragonfly larvae, whole prey fish, or axial muscle tissues) will then be freeze-dried or oven dried at 5°C until a nearly constant mass (change in dry mass less than one percent after 24 hours of additional drying) and final dry weight recorded (to 0.001 grams).

Taxonomy

Through a research laboratory GLKN may occasionally require that select dragonfly larvae be identified to species, or that a voucher collection be created and maintained. Either of these would be an additional expense; the necessity and affordability of this level of detail will be assessed when contract statements of work and/or annual contract modifications are developed. Additional dragonfly larvae beyond the 20 to be assessed for THg from each site will be collected when preservation of voucher specimens is desired. Most larvae present in the Great Lakes region can be identified to species with taxonomic keys by Hilsenhoff (1995) and Needham et al. (2000). Quality control for taxonomic identification of larval dragonflies should follow procedures outlined by Barbour et al. (1999), and questionable specimens should be taxonomically verified by a recognized dragonfly specialist. At a minimum it is highly desirable that an analytical laboratory be able to identify larval dragonflies down to *family*, to be able to check against those reported by field crews.

When fish are collected field crews will identify samples down to the lowest taxonomic level possible. However, it is highly desirable that an analytical laboratory be able to identify fish to *species*, to be able to check against those reported by field crews.

It will be a prerequisite for an analytical laboratory to verify the field crew identifications, or independently identify each sample to species, if necessary.

Laboratory Performance Evaluation

The ability to use environmental data to reveal long-term trends is enhanced with consistency in analytical methods and low variability in detection and reporting limits both within and among analytical laboratories. 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 certifications and/or federal programs in which they participate. Copies of relevant certifications for each analyte and/or method will be kept on file for each laboratory contracted for the duration of this monitoring effort.

The GLKN will assess the differences in detection and reporting limits, past performance evaluations, cost estimates, and other criteria such as relevant certifications, prior to selecting a contract laboratory. The laboratory selected by GLKN must be able to detect and report concentrations appropriately low such that changes in mercury concentrations can be detected early. The laboratory selected must have a rigorous QAP plan and meet the detection limits and performance criteria outlined below.

Laboratory Detection Limits and Analytical Methods for Mercury

The method used in calculating minimum detection limits (MDLs) and method limit of quantitation (MLOQ) or laboratory reporting limits (RL) may differ for each contract laboratory. Most laboratories routinely recalculate MDLs, MLOQs, and QC sample control limits using repeated measurement of standard samples or multiple percent recoveries on a quarterly or annual basis. MDLs and MLOQs are also a function of the analytical technology and individual instruments. Sample detection limits (DLs) and analytical methods for larval dragonfly and fish tissue samples collected from GLKN parks from 2008 to present are shown in Table 4. 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, and for data validation and certification.

Table 2. Detection limits and analytical methods for dragonfly larvae and fish tissue analyzed for mercury in Great Lakes Network parks from 2008–present. THg=total mercury, MeHg=methylmercury.

Period	Laboratory	Analyte	Tissue Type	Method Detection Limit	Method
2008–2012	University of Wisconsin-LaCrosse	THg	Larval dragonflies	5.0 ng/ga	EPA 1631
		THg	Whole prey fish	8.8 ng/ga	EPA 1631/ EPA 7473
		THg	Fish axial muscle tissue	12.1 ng/ga	EPA 1631/ EPA 7473
2015–2016	USGS CERL Lab – Corvallis, OR	THg	Fish axial muscle tissue	<1 ngb	EPA 7473
2008–2012	University of Wisconsin-LaCrosse	MeHg	Larval dragonflies	0.89 ng/ga	EPA 1630
2014–2015	Wisconsin State Lab of Hygiene	THg	Larval dragonflies	1.0 ng/ga	EPA 1631
2012–2015	University of Maine	THg	Larval dragonflies	<1.0 ngb	EPA 7473
2016–present	USGS CERL Lab – Corvallis, OR	THg	Larval dragonflies	<1.0 ngb	EPA 7473
2014–present	USGS CERL Lab – Corvallis, OR	MeHg	Larval dragonflies	<0.1 pgb	EPA 1630

^a Equivalent to parts-per-billion on a dry-weight basis

^b Total mass of THg or MeHg in sample

Based on the historic DLs shown in Table 4, analytical laboratories assessing THg in dragonfly larvae or fish will be required to achieve maximum MDLs of 1.0 ng/g and 10.0 ng/g, respectively. A prerequisite for selection of and contracting with an analytical laboratory will be the laboratory's ability to meet or exceed (i.e., lower) these MDLs. 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 (e.g., mg/kg or ppm and ng/g or ppb) for the same analytes. A checklist to be used in data validation is included in Attachment A.

Laboratory Performance Criteria

Comparability, representativeness, precision, systematic error, and completeness of data generated by contract laboratories will be estimated and documented according to this QAP during data validation. Unless otherwise justified (for example, to be consistent with State requirements), and in order to be consistent with past laboratory performance criteria for mercury monitoring in GLKN parks (Nelson et al. 2015, Wiener et al. 2016), in no case will Measurement Quality Objectives (MQOs) less stringent than those shown in Table 5 be acceptable. All MQOs will be used as data rejection criteria.

Table 5. Minimum precision and accuracy objectives by analyte and tissue type.

Analyte	Tissue Type	Precision Objective	Accuracy Objective
Total mercury	Larval dragonflies	± 10%	± 10%
	Fish	± 10%	± 10%
Methylmercury	Larval dragonflies	± 10%	± 15%
	Fish	± 10%	± 15%

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 A.

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.

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 the formula below. 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.

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

Determining required sample sizes and attendant completeness goals was done in a stepwise manner, considering desired statistical power and minimum detectable differences (see discussion in the protocol narrative).

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 MLOQs 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.

Laboratory Precision

In general, precision is the agreement of independent data resulting from repeated measurement under specified conditions (Taylor 1987). Measurement precision is 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 matrix specific precision and repeatability. Relative percent differences (RPDs) and relative standard deviations (RSDs) are calculated for laboratory duplicates and for samples analyzed more than twice. The RPD of these QC samples should fall within the control limits shown in Table 5, and is calculated as:

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

$$RSD = \left(\frac{StDev(A:B)}{Mean(A:B)} \right) \times 100$$

Where A is the first reading, and B is the last (second in the case of duplicates) reading.

Laboratory Accuracy

Accuracy (analytical bias) is estimated for each laboratory method through the analysis of certified reference materials (CRMs), liquid calibration standards (LCSs), and/or spiked samples (i.e., matrix spikes; MS). Spiked sample analyses are conducted by the contract laboratory to identify and assess matrix interference, which reduces recovery in specific sample types. A spiked sample consists of sample material with a known concentration of LCS added before any sample preparation procedures are carried out. The unmanipulated sample and an aliquot of the same mass spiked with a known quantity of mercury are then analyzed using normal procedures and the spike recovery is calculated as:

$$Spike\ Recovery = \left(\frac{MS - X}{Spike} \right) \times 100$$

Where MS is the mass (ng or pg) of mercury recovered in the matrix spike, X is the mass of mercury in the unspiked aliquot, and “Spike” is the mass of mercury added to the MS.

Laboratory blanks are used to estimate handling and analytical contamination during sample analysis.

The laboratory QAP 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 QAP. During data validation GLKN will ensure that the laboratory performance meets the precision and bias MQOs tabulated in Table 5.

Laboratory Instruments

Laboratory instruments such as those used to analyze samples for THg and MeHg are calibrated periodically and in accordance with manufacturer recommendations. Each laboratory specifies its own calibration procedures, though typical protocols include multiple point calibrations across the range of expected sample concentrations, analysis of method and instrument blanks, and analysis of LCSs and CRMs to verify calibration curves. In addition, each analytical run should include periodic analysis of blanks, CRMs, and LCSs throughout the run to ensure within-run QA/QC guidelines are met. Each laboratory should define these procedures in a QAP. Review of these procedures will be documented using the laboratory checklist in Attachment A.

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 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 the RPD and fraction of change:

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

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

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

Ideally, the average RPD will be within 5% of the mean of the old or the new method measurements. Acceptable RPDs will be agreed upon by GLKN and the contracted analytical lab. Corrective action for exceeding RPDs when the laboratory updates equipment or uses a new method involves comparing equipment or methodology over an extended period of time, and if necessary calculation and application of 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

These comparisons 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.

Accreditation Standards

The Great Lakes Network requires that cooperators, contractors and analytical laboratories are certified and/or accredited to ensure safety, when applicable, and quality of results. Table 6 lists the activities and requirements.

Table 6. Certification and accreditation requirements.

Activity Type	Activity	Parameter	Requirement	Location of Documentation ¹
Professional Permits and Certifications	Fish sampling	Handling and sampling fish	Institutional Animal Care and Use Committee (IACUC) permit.	Copies on file with the NPS IACUC and in the GLKN office
	Boat operations	Traveling to and from sampling sites	MOCC certification for NPS personnel operating boats	Web based certification maintained by DOI-NPS.

¹ If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

Although not shown as a required certification in Table 6, it is common for commercial analytical laboratories to be certified through the National Environmental Accreditation Program. However, many research analytical laboratories, while not necessarily certified through this program, have more stringent internal performance requirements and more flexibility in their use of instruments to maximize data quality and output, as opposed to the common one-size-fits-all approach that commercial laboratories often utilize. When we contract with an analytical laboratory for mercury analysis we will place emphasis on the laboratory's ability to supply satisfactory measurement quality objectives, prior experience, and documentation, rather than accreditation.

Records Management

Table 7 summarizes the QA/QC procedures related to records management. All records must be kept, from field activity through sample results. Metadata for all published data products must be FGDC compliant. The abstract for all metadata must include a link to the Integrated Resource Management Applications record for the protocol to ensure that data can be traced to all methods used for data collection, processing, the latest version of the protocol, this QAP, and all associated standard operating procedures. For more details on the GLKN's overall strategy for metadata generation, management, and distribution see Chapter 8, Data Documentation, of GLKN's Data Management Plan (Hart and Gafvert 2005) and the appendices of that document.

Data will be archived according to SOP #5, Data Entry and Management, for digital data, paper copies, and field forms. Field forms are maintained indefinitely. Summary reports 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 geometric mean, median, maximum and minimum values, and measures of variability (e.g., CV, standard error, variance). 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 #6, Data Analysis.

Table 7. Summary of QA/QC procedures pertaining to records management.

Procedure	Description
Project binder	Containing: checklist of QA/QC reminders, copies of decontamination, sample collection and processing SOPs, copies of equipment calibration and troubleshooting instructions, chain of custody forms, blank field forms.
Site binders	Containing: GPS coordinates for verification of correct sampling location, table of previous field measurements to compare with new measurements.
Field forms	Field forms are the only written record of field measurements. Place copies in site binders and keep originals on file indefinitely.
Electronic data entry	Approximately 10% of electronically-transferred data should be spot checked on a random basis for errors. If errors are found, another 10% are spot checked. 100% of manually-entered data will be checked by a second person.
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. Electronic records are maintained on a server that is backed up daily.
Field methods	Require consistent sampling methods.
Sample preservation	Environmental samples should be kept cold, as dry as possible, and frozen as soon as possible, after collection.
Chain-of-custody	A chain-of-custody includes not only the form, but all references to the sample in any form, document or log book which 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.

Table 7 (continued). Summary of QA/QC procedures pertaining to records management.

Procedure	Description
Laboratory methods	Require consistent analytical methods and detection limits.
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 Transfer

Import field and laboratory data into NPSTORET as soon as possible after receiving the data, according to SOP #5, Data Entry and Management. 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 for the same analyte. Follow SOP #5, Data Entry and Management, when importing data.

Contract laboratories should use the STORET-supported laboratory remark codes (Table 8) 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 here:

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

Table 8. STORET-supported laboratory remark codes.

Code	Definition
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

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.

Data Validation, Certification, and Usability

Data validation is the method in which data are proved 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 A. Data are validated by comparing the actual estimates for MDL and MLOQ, 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.

After data are returned to GLKN by WRD and appropriate documentation is completed, the NPSTORET datasets are officially considered certified. 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) they and their associated metadata 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. These procedures protect the integrity of the data and allow the history of each data record to be traced.

Note: as per IMD Data Certification Guidelines, in the absence of this QAP being approved, all data that have been through the QA/QC procedures as outlined in the approved protocol narrative and SOPs are deemed “acceptable.” After review and approval of this QAP, data certification will occur no less frequently than one year after data processing is complete.

Once data validation and certification are 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.

Roles and Responsibilities

Table 9 identifies organizations and individuals involved in the implementation of monitoring mercury in dragonfly larvae and fish, including all contractors and subcontractors.

Table 9. Roles, responsibilities, and contact information for persons or organizations affiliated with implementation of monitoring mercury in dragonfly larvae and fish.

Name	Affiliation / Position	Role	Contact Info
Carmen Thomson	Regional Program Manager	QAP approval, protocol approval and ensuring that data meet quality objectives.	402-661-1876; carmen_thomson@nps.gov
<currently vacant>	Network Program Manager	Program coordination.	715-682-0631 x221;
David VanderMeulen	Project Manager	Protocol implementation, data QA/QC and certification, data analysis, and reporting.	715-682-0631 x224; david_vandermeulen@nps.gov
Mark Hart	Data Manager	Oversight of the data management architecture.	715-682-0631 x226; mark_hart@nps.gov
Richard Damstra	Aquatic Ecologist	Primary data steward for the protocol, including data management, QA/QC, and data extraction.	715-682-0631 x238; richard_damstra@nps.gov
Alex Egan	Aquatic Ecologist	Secondary data steward for the protocol, including data management, QA/QC, and data extraction.	715-682-0631 x230; alex_egan@nps.gov
Randy Lehr	Professor, Northland College	Oversight of a field crew responsible for sampling larval dragonflies at five GLKN parks.	715-682-1261; rlehr@northland.edu
Brian Clary	Supervisor, Inorganic Chemistry, Wisconsin State Laboratory of Hygiene	Laboratory point of contact for mercury analyses of fish.	800-466-0403; Brian.clary@slh.wisc.edu
Sarah Nelson	Professor, University of Maine	Oversight of a laboratory focused on analysis of mercury in larval dragonflies.	207-949-4552; sarah.j.nelson@maine.edu
Colleen Flanagan-Pritz	Ecologist, NPS Air Resources Division	NPS point of contact for national dragonfly monitoring project.	303-969-2806; colleen_flanagan_pritz@nps.gov

Project Quality Assessment

Routine Data Quality Evaluations

Periodic data audits and quality control inspections will be conducted to maintain and improve data quality (Table 10). Audits will verify conformance with the data quality procedures specified in this plan and protocol SOPs. These quality checks are designed to promote continuous feedback and improvement of both the data and the quality planning process. Results of quality assessments will be documented and reported to the network program manager as an internal report with the project manager being responsible for ensuring that any nonconformities in data management practices are corrected.

Table 10. Routine data quality evaluation reports and checklists produced as a part of implementing the protocol for monitoring mercury in dragonfly larvae and fish.

Report/Checklist	Purpose & Description	Frequency
Data Quality Objectives	Ensures that data quality objectives are appropriate to meet stated monitoring objectives	During development of the QAP and at periodic protocol reviews (about every 5 years).
Internal Quality Assurance Reports	To assess sample size, missing values, data flags, and quality objectives relative to goals for analysis and reporting	Conducted cumulatively for the period of record for mercury and physical measurements of larval dragonflies and fish when data are certified. These precede all data analyses prior to publication.
Permits Checklist	Checklist that ensures appropriate permits and permit reporting is completed prior to field work	Prior to each field season

Communicating Data Quality

The protocol lead will reference this QAP and the appropriate Internal Quality Assurance Report when disseminating any data collected under this protocol, in addition to providing all data quality annotations with data.

Documentation

Records

NPS Director's Order 11D: Records and Electronic Records Management (2012) and its appendix, *NPS Records Schedule*, describe NPS activities and standards in maintaining and providing access to records at all levels of the Service. DO 11D states that all records of natural and cultural resources and their management are considered necessary for fulfillment of the NPS mission. The NPS Records Schedule provides guidelines on maintaining the results of Inventory and Monitoring Programs and indicates that all natural resource records are considered “permanent” and need to be retained either in an appropriate park museum facility or at the National Archives. Records associated with this protocol are shown in Table 11.

Table 11. Required (**bold**) and suggested records to be maintained as a part of implementing the protocol for monitoring mercury in dragonfly larvae and fish.

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Data collection field forms	Reference files and small data sets used for statistical analyses	Lab certifications and documented methods	Internal Quality Assessment Reports	Manuscript submittal forms for published reports
Permit application / acceptance memos	Personnel training certifications / documentation	Lab raw data electronic submittal files	Internal Memos	Peer review memos and author responses for published reports
Photos	–	Lab QA/QC reports	–	–

Information Dissemination and Reporting

All data reports disseminated for general (public) use will be based on certified data. In general, data will be certified prior to internal use for supporting park-, regional-, or national-level resource management decisions. However, in some cases the use of provisional data (data that have not undergone complete QA/QC as defined in this QAP) may be warranted to base decisions on best-available science. In addition, provisional data may be provided to internal and/or external science partners who are involved in data analysis and reporting, as a part of the data quality assurance process. Biostatisticians and partnering scientists must examine data closely during the analytical phase and will sometimes find data that need to be excluded from analysis or qualified as to its validity. Results from data evaluations by partners will be incorporated in the Internal Quality Assurance Reports.

Routine data summaries will occur annually for this monitoring. Summary reports will be produced periodically. Web-based visualization and summary tools for mercury data are under development (Colleen Flanagan-Pritz, pers. comm.). These tools and summary reports will be provided to park resource managers, park partners, and the public.

More comprehensive analyses of mercury trends will occur after a number of years of sampling. 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. Journal articles will follow Inventory and Monitoring Division peer review guidelines (NPS 2016) and those specific to the journal.

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Attachment A. Checklists for Selection of Laboratory and Validation of Laboratory Data.

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/MLOQ/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 _____

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 _____

The Department of the Interior protects and manages the nation's natural resources and cultural heritage; provides scientific and other information about those resources; and honors its special responsibilities to American Indians, Alaska Natives, and affiliated Island Communities.

NPS 920/147669, August 2018

National Park Service
U.S. Department of the Interior



[Natural Resource Stewardship and Science](#)

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