



Data Quality Standards for Monitoring Mercury in Resident Lake Fish – Version 1.0

Southwest Alaska Inventory and Monitoring Network

Natural Resource Report NPS/SWAN/NRR—2018/1731



ON THE COVER

Lake trout collected for mercury analysis from Kulik Lake in Katmai National Park and Preserve, Alaska.

NPS / EVAN BOOHER

Data Quality Standards for Monitoring Mercury in Resident Lake Fish – Version 1.0

Southwest Alaska Inventory and Monitoring Network

Natural Resource Report NPS/SWAN/NRR—2018/1731

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All manuscripts in the series receive the appropriate level of peer review to ensure that the information is scientifically credible, technically accurate, appropriately written for the intended audience, and designed and published in a professional manner.

This report is an accompanying component of the Southeast Alaska Inventory and Monitoring Network Environmental Contaminants protocol that received formal peer review by subject-matter experts who were not directly involved in the collection, analysis, or reporting of the data, and whose background and expertise put them on par technically and scientifically with the authors of the information. The information presented here is derived directly from the protocol.

Views, statements, findings, conclusions, recommendations, and data in this report do not necessarily reflect views and policies of the National Park Service, U.S. Department of the Interior. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the U.S. Government.

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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 these Data Quality Standards (DQSs) will promptly notify the Southwest Alaska Network (SWAN) project manager and/or 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 SOP 8 (Protocol Revision) in Bartz et al. 2018.

Revision History Log:

Previous Version #	Revision Date	Revision Author	Revision Description and Location in SOP	Justification for Revision	New Version #
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
–	–	–	–	–	–
Add rows as needed for each change or set of changes tied to an updated version number					

Acknowledgements

These DQSs were adapted from two similar documents published by the Southeast Alaska Network (SEAN; Sergeant 2018) and the Great Lakes Network (GLKN; VanderMeulen et al. 2018a). We wish to acknowledge the work of the authors, as well as their willingness to share that work. We also wish to thank several colleagues who improved this document with their reviews, including A. Miller, J. Lawler, C. Miller, and C. Flanagan-Pritz (National Park Service), and R. Gerlach and C. Furin (Alaska Department of Environmental Conservation).

Introduction and Summary

The purpose of this report is to document standards used by the Southwest Alaska Inventory and Monitoring Network (SWAN) for the collection, processing, storage, analysis, and publication of monitoring data, as described in the Protocol for Monitoring Mercury in Resident Lake Fish in SWAN parks (Bartz et al. 2018). The policies and procedures outlined in this abbreviated quality assurance plan complement the quality assurance plans for other monitoring activities conducted by the SWAN, and supplement the Quality Management Plan published by the National Park Service (NPS) Inventory and Monitoring Program (I&M) as a whole (in development). The plan also serves as a guide for all SWAN personnel involved in protocol-related activities and as a resource for identifying memoranda, publications, and other literature that describe associated techniques and requirements in more detail. Henceforth, this plan will be referred to as the Data Quality Standards (DQSs) for the protocol.

Protocol Overview

During the development of the SWAN Monitoring Plan (Bennett et al. 2006), resident lake fish were identified as a Vital Sign that warranted long-term monitoring, and contaminants were named as one possible metric. This protocol outlines a framework for monitoring mercury (Hg) concentrations in resident lake fish. It is adapted from a broader protocol for monitoring Hg in aquatic organisms, developed by the Great Lakes Network (GLKN; VanderMeulen et al. 2018b). The primary goal of this monitoring is to understand the spatial differences, temporal trends, and health ramifications of Hg contamination in resident lake fish from the SWAN. Three objectives will be addressed by this monitoring:

1. assess spatial differences in Hg contamination of resident lake fish, among waterbodies;
2. evaluate temporal trends in Hg contamination of resident lake fish, within waterbodies; and
3. identify waterbodies where Hg contamination of resident lake fish may pose a risk to humans and top predator piscivores by surpassing relevant toxicological benchmarks.

For each of these objectives, our monitoring will rely on total Hg in fish axial muscle as an indicator of methyl Hg exposure. It will target four high priority lakes which vary in size, depth, wetland cover, glacial influence, and Hg concentration. The focal species will be lake trout, a widespread, long lived, top predator in SWAN lakes. In short, the SWAN will sample 20 lake trout from all 4 lakes, once every 5 years.

Two broad types of data will be generated from this protocol. The first type includes observations and measurements that are recorded on field forms while sampling and processing fish, as well as photographs and coordinates that are taken. The second type includes results of analyses performed by contract laboratories. We will use NPSTORET as the primary data entry tool and data transfer mechanism.

Protocol Activities

Data are collected under this protocol during the course of five types of activities (Table 1), including:

1. Field Observation. Field crews will document site characteristics for each monitoring location throughout each sampling event. Standard site information – such as the date, time, park, waterbody, latitude, longitude, and crew member and data recorder names and affiliations – will be recorded. Supporting information also will be recorded, including lures used and bycatch counts by species.
2. Specimen Collection and Processing. The target sample size is 20 fish per lake, and the target fish species is lake trout. Fish will be caught via angling or gill nets, following state and federal fishing regulations. Once caught, fish will be screened for external lesions or other abnormalities, and then held in a net pen underwater or in a portable cooler on ice. Prior to departure, each fish will be euthanized, placed in a labeled plastic bag, and stored under refrigeration until dissection. Fish will be processed within 24 hours of euthanization. Each fish will be photographed, weighed, measured, sexed, and dissected by removing sagittal otoliths and axial muscle tissue. Specifically, we will remove three tissue subsamples per fish, with each subsample weighing ~10 g. Triplicate subsamples will be placed in separate bags and stored in a -20°C freezer. Otoliths will be stored in vials at room temperature.
3. Laboratory Analysis. The SWAN will rely on qualified contractors or government laboratories that meet monitoring program specifications and nationally accepted standards to perform laboratory analyses. Tissue samples will be shipped on dry ice to an analytical laboratory for Hg analyses. At the laboratory, tissue samples will be thawed gradually at room temperature. 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. Total Hg (THg) analysis will follow EPA Method 7473 (SW-846) and be reported as µg/g dry weight (dw). The moisture content of each sample will also be recorded to enable conversion of THg to wet weight (ww). Otoliths will be shipped separately to facilities with expertise in age analysis, where they will be set, sectioned, and mounted on microscope slides prior to reading, as described in Campana et al. 2008.
4. Data Derivation. Some values will be derived from raw field and lab data. For example, raw fish weights will be recorded three times and a mean weight will be derived. Likewise, descriptive statistics for THg concentration will include mean, median, maximum and minimum values, as well as skewness, kurtosis, and measures of variability. Data derivation also covers the calculation of covariates of interest.
5. Quality Assurance/Quality Control (QA/QC). SWAN staff will follow QA/QC procedures outlined in this document, as well as the SOPs associated with the monitoring protocol. Laboratories under contract for this protocol are required to have an approved laboratory Quality Assurance Plan (QAP), and to provide QA/QC data on results for every batch of samples.

Table 1. Activity matrix for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Activity Type	Activity #	Activity	Description
Field Observation	1	Site characterization	Data recorded during the course of this activity include: the date(s) and time(s) when fish were sampled; the park and lake names; the general location where fishing occurred; the names and affiliations of the data recorder and crew members; counts and species of bycatch; and general information (e.g., which lures worked best). See the field form provided in Appendix 3.1 of SOP 3.
Specimen Collection and Processing	2	Lake trout collection and processing	Data recorded during the course of this activity include: fish ID number; date, latitude, and longitude when caught; fork length, total length, weight, and gender; number of otoliths removed, qualitative description of stomach contents; and any general comments about the fish. Each fish is also photographed before dissection. See SOP 3 for additional details.
Laboratory Analysis	3	Lake trout tissue and otolith analysis	Data recorded during the course of this activity include: THg concentration ($\mu\text{g/g}$ dw and ww) and moisture content (%) in muscle tissue; fish age (yrs). See SOP 3 for additional details.
Data Derivation	4	Derived data	Data derived during the course of this activity include: mean fish weight; mean, median, maximum and minimum THg by lake; skewness, kurtosis, and measures of variability (e.g., coefficient of variation, standard error, standard deviation, and variance) for THg by lake; covariates of interest such as sockeye salmon spawner density (fish/km or ha), % wetland cover, and % glacier cover for a given lake.
Quality Assurance/Quality Control	5	QA/QC data	Data recorded during the course of this activity include: THg concentration in analytical and procedural blanks, and THg concentration in replicate samples.

Sampling Design

Information regarding the sampling design is provided in Table 2, divided by the activity types listed in Table 1. Only those activity types in which data are collected routinely according to fixed designs are included in the table. For additional specificity regarding spatial and temporal aspects of the sampling design, see the protocol narrative and SOP 3.

Table 2. Sample design matrix for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Activity Type	Activity #	Activity	Sampling Design	Revisit Design
Field Observation	1	Site characterization	Targeted; sampling sites are limited to four “Tier 1” lakes within two SWAN parks (Lake Clark National Park and Preserve and Katmai National Park and Preserve). These lakes were previously deemed as a high priority for freshwater monitoring (Shearer et al. 2015).	Revisit every five years
Specimen Collection and Processing	2	Lake trout collection and processing	Targeted; sampling sites are limited to four “Tier 1” lakes within two SWAN parks (Lake Clark National Park and Preserve and Katmai National Park and Preserve). These lakes were previously deemed as a high priority for freshwater monitoring (Shearer et al. 2015).	Revisit every five years

Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements that clarify the objectives of observations, define the appropriate types of data, and specify the 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. DQOs include data quality values, which are qualitative, and measurement quality objectives and performance standards, which are quantitative.

Data Quality Values

Data quality values are the desired qualitative characteristics of datasets. Seven of these characteristics are addressed here: accuracy, representativeness, comparability, timeliness, completeness, consistent representation, and security (Table 3). Measurement quality objectives and performance standards are provided in the subsequent section.

Table 3. Data quality values for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Data Quality Category	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, mass, 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 Hg in resident lake 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, location of sampling, and sample handling are taken into account to ensure that the 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.	Before each sampling event, the project leader will ensure that field personnel are properly trained to collect and handle specimens, as outlined in SOPs 2 and 3. Subsequently, samples at each lake will be collected and analyzed using identical techniques. Laboratory analysis of THg will follow a nationally accepted protocol set forth by the U.S. Environmental Protection Agency (EPA). Resulting data will be comparable to data collected by other organizations.
	Timeliness / Currency	How recent the data need to be to be considered valid for their intended use. Data represent conditions and/or are 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 will be provided upon request, or if there is a risk to human health. Final 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, issues with laboratory equipment, or some other unforeseen loss of samples (e.g., an undelivered package) may result in missing values, particularly for otoliths. Data analysis procedures are available to handle missing values.

Table 3 (continued). Data quality values for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Data Quality Category	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	THg concentrations will be reported in units of $\mu\text{g/g dw}$, which can be converted to units of $\mu\text{g/g ww}$. These concentrations will be compared to four published toxicological benchmarks ($0.300 \mu\text{g/g ww}$ from Borum et al. 2001; $0.20 \mu\text{g/g ww}$ from Hamade 2014; $0.050 \mu\text{g/g ww}$ from GLFAW 2007; and $0.500 \mu\text{g/g ww}$ from Sandheinrich and Wiener 2011). Because these thresholds may change as knowledge advances, periodic reporting of monitoring results will clearly define the most recent thresholds and their sources.
Data Accessibility	Security	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 EPA's Water Quality Exchange. At present, we know of no potentially protected data that will be generated from this monitoring. Networks must identify and ensure that potentially protected data, including sensitive species location data, sensitive cultural resource information, and other data as protected by DO#66 are not released to the public. Protected data may be released in limited cases only with park superintendent approval following park procedures.

Measurement Quality Objectives – Field Work

Measurement quality objectives are data quality requirements specifying the levels of precision, bias, sensitivity, resolution, etc. that data must meet to be accepted for use in analysis. Table 4 focuses on measurement quality objectives pertaining to field work. Measurement quality objectives for laboratory analyses are specified in Table 5.

Table 4. Measurement quality objectives for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Activity	Measure / Quality Indicator	Quality Objective
Field Observation	Location Accuracy	GPS coordinates listed in the field form should represent the general locations where fishing occurred (e.g., the midpoint along a linear shoreline transect where samples were collected). If fish are caught >1 kilometer (km) apart, distinct coordinates should be recorded.
Specimen Collection and Processing	Identification Resolution	Taxonomic identifications should be made to the species level.
	Identification Accuracy	Taxonomic identifications, performed by a field crew lead with documented identification proficiency, should be 100% accurate. DNA from fin clips may be used to confirm identifications, if necessary.
	Identification Completeness	100% of taxonomic identifications should be made to the species level.
	Fish Length Accuracy	Total length and fork length should be measured to the nearest millimeter (mm), with an accuracy of +/- 5 mm.
	Fish Weight Accuracy	Fish weight should be measured to the nearest gram (g) if the scale allows, with an accuracy of +/- 10 g.
	Fish Gender Accuracy	It is not always possible to determine lake trout gender in the field. A category of "U" (for unknown) is provided on the field form.
	Fish Diet Accuracy	Stomach contents are intended as a qualitative description, to quickly summarize what an individual fish ate recently. Stomach contents should not be enumerated to the species level.
Laboratory Analysis	MDL, Bias, Precision, Completeness ¹	See lab method performance requirements for specifications by analyte (Table 5).

¹ MDL = Minimum Detection Limit, defined as 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.

Measurement Quality Objectives – Laboratory Analyses

Performance Standards

Performance standards are data quality requirements specifying the sensitivity, variability, detection ability, etc., of equipment to reliably measure characteristics of target populations. The ability to use environmental data to reveal long-term trends is enhanced with consistency in analytical methods and performance standards, within and among laboratories.

Selecting Laboratories

The SWAN will assess differences in performance standards, cost estimates, and relevant certifications, prior to selecting a laboratory. The laboratory selected must have a rigorous quality assurance plan (QAP) and meet the performance standards outlined below (Table 5). A checklist for selecting laboratories and documenting their compliance with QA/QC expectations is included in Appendix A.

Table 5. Performance standards by analyte for the SWAN protocol for monitoring mercury in resident lake fish (Bartz et al. 2018).

Analyte	Units	MDL Objective ¹	Maximum Allowable Bias ²	Maximum Allowable Precision ³	Completeness Objective ⁴
THg ⁵	µg/g dw	< 0.01 µg/g dw	≤ 10% from a standard reference material	Triplicate runs of a subset of samples must have a CV < 10%	95%
Age	years	1 year	"True" value is unknown	Duplicate runs of 15% of samples must have a RPD of < 5%	95%

¹ MDL = Minimum Detection Limit, as defined in the footnote for Table 4.

² Bias goals are expressed as a percent deviation from a "true" value.

³ Precision goals are expressed as relative percent difference (RPD).

⁴ Completeness goal is the percentage of expected results that are obtained successfully.

⁵ THg will be analyzed using EPA Method 7473.

It is common for commercial laboratories to be certified through the National Environmental Accreditation Program. However, many research 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 selecting a laboratory for THg or age analysis we will place emphasis on the laboratory's ability to supply satisfactory measurement quality objectives, prior experience, and documentation, rather than accreditation.

Changing Methods or Laboratories

When a change in methods is made by a contracted 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 relative percent difference (RPD) and fraction of change (FOC), for each sample:

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

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

where *new* = data from the replacement method or lab, and *old* = data from the original method or lab. Ideally, the average RPD will be <5%. Acceptable RPDs will be agreed upon by the SWAN and the contracted laboratory. Corrective action for RPDs >5% involves comparing equipment or methodology over an extended period of time, and if necessary calculation and application of the FOC. 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 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 contaminant concentrations in fish.

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Appendix A

Checklist for Laboratory Selection

Laboratory: _____

Address: _____

Contact person: _____

_____ Received a copy of quality assurance plan (QAP)

_____ Received a copy of certifications

_____ Received a list of analytical methods used

_____ Define the limits of detection (LOD) and limit of quantitation (LOQ) 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 inter-laboratory participation documentation

_____ Sample handling log-in and chain-of-custody (COC) are documented in the QAP

_____ Equipment maintenance and calibration procedures are documented in the QAP

_____ Internal QA/QC documented in the QAP

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: _____

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