



Data Quality Standards for Environmental Contaminants Monitoring in the Southeast Alaska Network

Version EC-2018.1

Natural Resource Report NPS/SEAN/NRR—2018/1770



ON THE COVER

Examining a Dolly Varden from the Taiya River before preserving for mercury analysis
Photograph courtesy of Andrew Cyr (University of Alaska Fairbanks)

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Natural Resource Report NPS/SEAN/NRR—2018/1770

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Executive Summary

The purpose of this report is to document the standards used by the National Park Service (NPS) Southeast Alaska Network (SEAN) for activities related to the collection, processing, storage, analysis, and publication of monitoring data for environmental contaminants as described in the Environmental contaminants monitoring protocol for the National Park Service, Southeast Alaska Inventory and Monitoring Network: Version EC-2018.1 (Sergeant et al. 2018). The policies and procedures documented in this quality assurance plan complement the quality assurance methods for other monitoring activities conducted by the SEAN. The plan also serves as a guide for all SEAN personnel who are involved in protocol/program activities and as a resource for identifying memoranda, publications, and other literature that describe associated techniques and requirements in more detail and as a resource for end-users of program data.

Introduction

The purpose of this report is to document the standards, used by the Southeast Alaska Inventory and Monitoring Network (SEAN) for activities related to the collection, processing, storage, analysis, and publication of monitoring data as described in the Environmental Contaminants Monitoring Protocol for the SEAN (Sergeant et al. 2018). The policies and procedures documented in this quality assurance plan for activities are consistent with other monitoring activities conducted by the SEAN and national program guidance. The plan also serves as a guide for all SEAN personnel who are involved in protocol/program activities and as a resource for identifying memoranda, publications, and other literature that describe associated techniques and requirements in more detail.

Protocol Overview

Airborne, freshwater, and marine contaminants were designated as priority long-term monitoring programs (vital signs) for the SEAN. In the protocol (Sergeant et al. 2018), these three vital signs are integrated into one environmental contaminants monitoring program in order to increase data collection and reporting efficiency for tightly connected ecosystem components that are exposed to a common suite of pollutants.

The goal of the environmental contaminants monitoring program is to monitor a suite of priority pollutant or contaminant threats to the parks of Southeast Alaska. This will be accomplished by implementing two primary priorities. First, this protocol will produce a long-term surveillance record of priority airborne, freshwater, and marine pollutants or contaminants in sentinel species at select sites in SEAN parks to enable comparisons with future conditions. Second, the protocol establishes a means to compare pollutant and contaminant concentrations against state, federal, or other relevant criteria for human and ecological health, as a means of evaluating the potential for impairment of NPS resources.

The three specific program objectives are:

- 1) Monitor lichen tissue concentrations for nitrogen, sulfur, and 25 additional elements in conformance with protocols developed for the Tongass National Forest
- 2) Monitor total mercury in riverine Dolly Varden tissue using existing Environmental Protection Agency (EPA) protocols
- 3) Monitor a broad suite of metals, polycyclic aromatic hydrocarbons (PAHs), and persistent organic pollutants (POPs) in bay mussels, in conformance with national protocols developed by the National Oceanic and Atmospheric Administration (NOAA) Mussel Watch Program.

Protocol Activities and Modules

Data are collected or derived as a part of the Environmental Contaminants Monitoring Protocol for the SEAN (Sergeant et al. 2018) in four different activities or modules (**Error! Reference source not found.**).

1. Site Recon, Establishment, and Maintenance. Data associated with sampling locations (descriptions, locational data, navigation, surveying data) and/or maintenance logs associated with monitoring station infrastructure.
2. Field Data Collection. Measurements taken and recorded by IMD or cooperator staff. These should often be broken into activities based on implementation timing and/or scale of measurement (data collected in transects are a different activity than plot data).
3. Field Sample Data. Information related to samples collected that will be processed and analyzed by NPS staff. This may include specimens, vouchers, or lab data.
4. Lab Sample Data. Sample collection and preparation information, shipping and chain of custody information, and resultant data and QA/QC information provided upon delivery from the lab.

Table 1. Protocol activity matrix for the SEAN Environmental Contaminants Monitoring Protocol (Sergeant et al. 2018).

Module	Activity Number	Activity	Description
Site Recon, Establishment, and Maintenance	1	Site Recon	Investigate suitability of sites to be used as sampling location. Data collected and retained (descriptions, photos) for reference when being considered for future work or work to be conducted as a part of other monitoring efforts.
Field Observations	2	Lichen collection	Plot name, date, time, collector, species, moisture status, and substrate; see field sheet and further description in SOP 2
	3	Dolly Varden trap data	Site name, trap number, depth, date set, time set, date retrieved, time retrieved, species, number caught; see field sheet and further description in SOP 3
	4	Dolly Varden individual fish data	Site name, trap number, serial ID, species, fork length, mass, kept for analysis (y/n); this data sheet cross-references with trap data sheet; see further description in SOP 3
	5	Bay mussel collection	Site ID, time of collection, water temperature, height of collection, GPS location, salinity sample, number mussels collected; see field sheet and further description in SOP 4
Field Sample & Specimen Collection and Processing	6	Lichen specimen collection	Tissue collected by hand and returned to office for drying and preparation for shipping to contracted laboratory in accordance with SOP 2
	7	Dolly Varden specimen collection	Species collected by baited minnow trap, euthanized, and kept cool until shipping to contracted laboratory in accordance with SOP 3
	8	Bay mussel specimen collection	Species collected by hand and kept cool until shipping to contracted laboratory in accordance with SOP 4
Laboratory Data (also see Table 3.1 in Sergeant et al. 2018)	9	Lichen tissue chemistry	Analysis of total nitrogen, total sulfur, and 25 additional elements
	10	Dolly Varden tissue total mercury	Fish tissue preparation and total mercury analysis
	11	Bay mussel tissue chemistry	Mussel tissue preparation and analysis of organics, trace metals, polycyclic aromatic hydrocarbons, poly chlorinated biphenyls, and organochlorines

Sampling Design

Information regarding the sampling design is provided in **Error! Reference source not found.**

Table 2. Activity-level sample design matrix for the SEAN Environmental Contaminants Monitoring Protocol (Sergeant et al. 2018).

Module	Activity Number	Activity	Sampling Design	Revisit Design
Site Recon, Establishment, and Maintenance	1	Site recon	Targeted; across the three SEAN parks, site selection is driven by exposure to potential contaminants sources, reliable foot or boat access, and co-location with existing vital signs monitoring efforts. There are six lichen sites, six Dolly Varden sites, and seven bay mussel sites. See Section 2 of Sergeant et al. (2018) for more detailed rationale and site descriptions. It is important to note that this is a surveillance program designed to track whether priority pollutants cross defined concentration thresholds that may pose a risk to the generally pristine condition of Southeast Alaska parks. We do not expect to detect contaminants trends in this program.	Revisit every five years
Field Observations	2	Lichen collection	Targeted; across the three SEAN parks, site selection is driven by exposure to potential contaminants sources, reliable foot or boat access, and co-location with existing vital signs monitoring efforts. There are six fixed lichen sites	Revisit every five years
	3,4	Dolly Varden trap and individual fish data	Targeted; across the three SEAN parks, site selection is driven by exposure to potential contaminants sources, reliable foot or boat access, and co-location with existing vital signs monitoring efforts. There are six fixed Dolly Varden sites	Revisit every five years
	5	Bay mussel collection	Targeted; across the three SEAN parks, site selection is driven by exposure to potential contaminants sources, reliable foot or boat access, and co-location with existing vital signs monitoring efforts. There are seven fixed bay mussel sites.	Revisit every five years

Data Quality Objectives

Data quality values and standards for implementation are provided in Table 3 through **Error! Reference source not found.**

Table 3. Data Quality Values (DQVs) for the SEAN Environmental Contaminants Monitoring Protocol (Sergeant et al. 2018).

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.	Field collections of specimens follow the methods described in SOPs 2-4 of Sergeant et al. (2018); Laboratory methodologies follow the individual methodologies cited within Table 3.1 of Sergeant et al. (2018).
	Representativeness	Measurements represent conditions at the time of sampling. Combined with accuracy, leads to repeatable data collection.	Drivers of contaminant concentrations originate from local to long-range sources (Table 1.2 of Sergeant et al. 2018). The scales of inference across this environmental contaminants protocol can range from hundreds of meters (e.g. bay mussels exposed to an oil spill) to thousands of kilometers (e.g. wet deposition Hg transported across the Pacific Ocean). Measurements are general indicators of contaminants conditions within park boundaries.
Contextual Data Quality	Comparability	The degree to which data can be compared among sample locations, data sources, or periods of time.	Samples at each site within the SEAN network will be collected and analyzed using identical techniques. Laboratory analyses follow nationally accepted protocols set forth by the Environmental Protection Agency, Forest Service, and National Oceanic and Atmospheric Administration (Table 3.1 of Sergeant et al. 2018). In many cases, these data will be comparable to contaminants data collected by other organizations. Before each sampling event, the project leader will need to ensure that individuals are properly trained to collect and handle specimens according to SOPs 2-4 (Sergeant et al. 2018).

Table 4 (continued). Data Quality Values (DQVs) for the SEAN Environmental Contaminants Monitoring Protocol (Sergeant et al. 2018).

Category	Data Quality Value	Definition	Protocol Considerations
Contextual Data Quality (continued)	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.	At this time, no known contaminants risks have been identified within Southeast Alaska national park units. This program is designed to act as surveillance in the event that contaminants levels change and need to be examined with greater frequency and spatial resolution. The baseline data here also act as reference points in the case of a potential catastrophic even such as a large oil spill. Based on pilot work completed before this protocol, laboratory analyses will generally be available within one year after tissue collection.
	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.	Methods, sampling plans, and analyses are designed and implemented such that they result in a complete dataset across space and the planned period of record. Note that this value is for a collection of data records; how many measures are deemed valid and suitable for use at the completion of all QA/QC procedures.
Representational Data Quality	Consistent Representation	Use of standard definitions when describing data quality or resource quality based on data	Contaminant concentrations will be reported according the units prescribed in Table 3.1 of Sergeant et al. (2018). These concentrations will be compared to published thresholds of human health or environmental concern developed by the Forest Service and Environmental Protection Agency. We note that thresholds do not exist for all analytes and are subject to change based on new knowledge. To counteract this, periodic reporting of monitoring results will clearly define the most recent thresholds and their source.
Data Accessibility	Secure	Access to data, products, and systems limited to appropriate audiences.	At the time of drafting this document, we know of no potentially protected data that will be generated from this program. 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.

Table 5. Measurement Quality Objectives for the SEAN Environmental Contaminants Monitoring Protocol (Sergeant et al. 2018).

Measure	Quality Indicator	Quality Objective
Site Recon	Location Accuracy	GPS coordinates listed for each sampling site in Sergeant et al. (2018) are general guidelines and do not require field collectors to use accurate locations. The sampling priority is to collect target biota (lichen, Dolly Varden, bay mussels) within a reasonable distance (< 1 km) from the given coordinates. Habitat conditions will change over time and require collectors to range around sites for samples.
Field observations and collections	Taxonomic Resolution	Identifications made to the species level
	Identification Accuracy	100% accuracy (in extreme cases for Dolly Varden, DNA may be used to confirm identification)
	Identification Completeness	100% of observations made to species level
	Dolly Varden fork length accuracy	+/- 5 mm
	Dolly Varden mass accuracy	+/- 5 g
	Bay mussel water temperature accuracy	+/- 0.5°F (NOAA Mussel Watch program uses standard units)
Laboratory analysis	MDL, Bias, Precision, completeness	See lab method performance requirements for specifications by organism and analyte

Table 5. Laboratory method performance requirements and analysis methods to be used for lichen tissue analysis. Citations or descriptions for each method can be found in Table 3.1 of Sergeant et al. (2018). For an extended summary of laboratory methods and individual analyte performance, please refer to Appendix A of the DQS.

Analyte	Units	MDL Objective	Maximum Allowable Bias ¹	Maximum Allowable Precision ²	Completeness Objective ³
Total nitrogen	% dry weight	0.032%	No specific thresholds have been identified for bias of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	No specific thresholds have been identified for precision of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	95%
Total sulfur	% dry weight	0.033%	No specific thresholds have been identified for bias of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	No specific thresholds have been identified for precision of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	95%
25 additional elements (Al, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, Si, Sr, Ti, V and Zn)	µg/g dry	Range from 0.001 (Be, Li, Mo) to 0.258 (K)	No specific thresholds have been identified for bias of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	No specific thresholds have been identified for precision of lichen tissue analyses, but labs can provide analytical blank values and run duplicate samples to assess sample variability	95%

¹ Bias goals are expressed as either absolute difference (+/- value) or percent deviation from a “true” value.

² Precision goals are expressed as relative percent difference (RPD) or relative standard deviation (RSD) between two or more replicate measurements.

³ Completeness goal is the percentage of expected results that are obtained successfully

Table 6. Example laboratory method performance requirements and analysis methods to be used for Dolly Varden tissue total mercury analysis. Citations or descriptions for each method can be found in Table 3.1 of Sergeant et al. (2018).

Analyte	Units	MDL Objective	Maximum Allowable Bias ¹	Maximum Allowable Precision ²	Completeness Objective ³
Total mercury	ng/g wet	12.5	≤ 20% from a standard reference material	Triplicate runs of each sample must have CV < 10%	95%

¹ Bias goals are expressed as either absolute difference (+/- value) or percent deviation from a “true” value.

² Precision goals are expressed as relative percent difference (RPD)

³ Completeness goal is the percentage of expected results that are obtained successfully

Table 7. Example laboratory method performance requirements and analysis methods to be used for bay mussel tissue chemistry. Citations or descriptions for each method can be found in Table 3.1 of Sergeant et al. (2018). For an extended summary of laboratory methods and individual analyte performance, please refer to Appendix B of the DQS.

Analyte	Units	MDL Objective	Maximum Allowable Bias ¹	Maximum Allowable Precision ²	Completeness Objective ³
Trace metals	µg/g dry	Range from 0.00005 (Hg) to 0.223 (Al)	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.
PAHs	ng/g dry	Range from 0.320 (Dibenzothiophene) to 24.9 (C2,3,4-Naphthalenes)	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.
OC/PCBs	ng/g dry	Range from 0.21 (Oxychlorane; Cis-Nonachlor; PCB 187) to 0.60 (12 different PCB congeners)	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.	Due to the large number of analytes included within each general group of contaminants, please refer to Appendix B for an extended QA/QC summary.

¹ Bias goals are expressed as either absolute difference (+/- value) or percent deviation from a “true” value.

² Precision goals are expressed as relative percent difference (RPD)

³ Completeness goal is the percentage of expected results that are obtained successfully

Table 8. Taxonomic standards to be used in Sergeant et al. (2018).

Module	Activity Number	Activity	Standard to be Used	Reference / Authority
Field Sample & Specimen Collection and Processing	6	Lichen specimen collection (<i>Hypogymnia enteromorpha</i> , <i>Hypogymnia inactiva</i> , and <i>Platismatia glauca</i>)	Field Subject Matter Expert	Field crew lead w/ documented identification proficiency
	7	Dolly Varden specimen collection (<i>Salvelinus malma</i>)	Field Subject Matter Expert; Scientific publication	Field crew lead w/ documented identification proficiency (Dolly Varden can be difficult to ID relative to other salmonids in the same system; under extreme circumstances, DNA analysis may be used to confirm species); Pollard et al. 1997
	8	Bay mussel specimen collection (<i>Mytilus trossulus</i>)	Field Subject Matter Expert	Field crew lead w/ documented identification proficiency

Literature Cited

- Pollard, W. R., G. F. Hartman, C. Groot, and P. Edgell. 1997. Field identification of coastal juvenile salmonids. Harbour Publishing, British Columbia, Canada.
- Sergeant, C. J., M. R. Bower, A. Cyr, and W. F. Johnson. 2018. Environmental contaminants monitoring protocol for the National Park Service, Southeast Alaska Inventory and Monitoring Network: Version EC-2018.1. Natural Resource Report NPS/SEAN/NRR—2018/1768. National Park Service, Fort Collins, Colorado.

Appendix A: Quality assurance/quality control measures for analysis of lichen tissue under the U.S. Forest Service National Air Quality Monitoring Program

(provided by University of Minnesota Research Analytical Lab)

Table A1. Analytical reporting units.

Matrix	Total S or N	25 other elements
Tissue	% present in tissue	µg /dry g

Table A2. Elements Method Detection Limits.

Elements	Tissue
N	< 0.032%
S	< 0.033%
Al	< 0.094%
As	< 0.013%
B	< 0.006%
Ba	< 0.01%
Be	< 0.001%
Ca	< 0.067%
Cd	< 0.002%
Co	< 0.006%
Cr	< 0.016%
Cu	< 0.007%
Fe	< 0.011%
K	< 0.258%
Li	< 0.001%
Mg	< 0.18%
Mn	< 0.013%
Mo	< 0.001%
Na	< 0.043%
Ni	< 0.003%
P	< 0.048%
Pb	< 0.064%
Rb	< 0.08%
S	< 0.045%
Si	< 0.087%
Sr	< 0.01%

Elements	Tissue
Ti	< 0.006%
V	< 0.009%
Zn	< 0.015%

Appendix B: Quality assurance/quality control measures for analysis of bay mussel tissue under the NOAA Mussel Watch Program

(provided by Juan Ramirez, Environmental Laboratory Manager, TDI-Brooks International, Inc.)

Table B1. Analytical reporting units.

Matrix	PAH	OC/PCBs	PBDE/PBB	BT	Trace Metals
Tissue	ng/dry g	ng/dry g	ng/dry g	ng/Sn dry g	ug/dry g

Table B2. Trace metals method detection limits. Sample size = 1.00g.

Metals	Tissue (Total µg)
Ag	0.00104
Al	0.10
As	0.00519
Ba	0.01
Cd	0.00104
Cr	0.00519
Cu	0.052
Fe	0.1
Hg	0.00005
Li	0.0207
Mn	0.021
Ni	0.0207
Pb	0.00519
Se	0.0104
Sn	0.00207
Zn	0.021

Table B3. PAH method detection limits. Sample size = 2.11g.

PAH	Tissue MDLs (ng/dry g)
cis/trans Decalin	6.55
C1-Decalins	13.1
C2-Decalins	13.1
C3-Decalins	13.1
C4-Decalins	13.1
Naphthalene	12.4
C1-Naphthalenes	0.690
C2-Naphthalenes	24.9
C3-Naphthalenes	24.9
C4-Naphthalenes	24.9
Benzothiophene	0.825
C1-Benzothiophenes	1.65
C2-Benzothiophenes	1.65
C3-Benzothiophenes	1.65
C4-Benzothiophenes	1.65
Biphenyl	3.18
Acenaphthylene	1.08
Acenaphthene	3.99
Dibenzofuran	1.34
Fluorene	1.05
C1-Fluorenes	2.10
C2-Fluorenes	2.10
C3-Fluorenes	2.10
Carbazole	2.04
Anthracene	0.813
Phenanthrene	1.88
C1-Phenanthrenes/Anthracenes	0.753
C2-Phenanthrenes/Anthracenes	2.63
C3-Phenanthrenes/Anthracenes	2.63
C4-Phenanthrenes/Anthracenes	2.63
Dibenzothiophene	0.320
C1-Dibenzothiophenes	0.874
C2-Dibenzothiophenes	0.640
C3-Dibenzothiophenes	0.640
C4-Dibenzothiophenes	0.640
Fluoranthene	1.41
Pyrene	0.669
C1-Fluoranthenes/Pyrenes	2.07

PAH	Tissue MDLs (ng/dry g)
C2-Fluoranthenes/Pyrenes	2.07
C3-Fluoranthenes/Pyrenes	2.07
C4-Fluoranthenes/Pyrenes	2.07
Naphthobenzothiophene	0.595
C1-Naphthobenzothiophenes	1.19
C2-Naphthobenzothiophenes	1.19
C3-Naphthobenzothiophenes	1.19
C4-Naphthobenzothiophenes	1.19
Benz(a)anthracene	0.325
Chrysene/Triphenylene	0.714
C1-Chrysenes	1.43
C2-Chrysenes	1.43
C3-Chrysenes	1.43
C4-Chrysenes	1.43
Benzo(b) fluoranthene	0.477
Benzo(k,j) fluoranthene	0.888
Benzo(a) fluoranthene	0.888
Benzo(e) pyrene	0.822
Benzo(a) pyrene	0.889
Perylene	5.05
Indeno (1,2,3-c,d) pyrene	1.26
Dibenzo (a,h)anthracene	1.11
C1-Dibenzo (a,h) anthracenes	2.23
C2-Dibenzo (a,h) anthracenes	2.23
C3-Dibenzo (a,h) anthracenes	2.23
Benzo (g,h,i) perylene	0.737
2-Methylnaphthalene	0.611
1-Methylnaphthalene	0.654
2,6-Dimethylnaphthalene	1.31
1,6,7-Trimethylnaphthalene	1.60
1-Methylfluorene	1.54
4-Methyldibenzothiophene	1.16
2/3-Methyldibenzothiophene	1.16
1-Methyldibenzothiophene	1.16
3-Methylphenanthrene	1.40
2-Methylphenanthrene	1.40
2-Methylantracene	1.40
4/9-Methylphenanthrene	1.40
1-Methylphenanthrene	1.40

PAH	Tissue MDLs (ng/dry g)
3,6-Dimethylphenanthrene	2.10
Retene	1.62
2-Methylfluoranthene	1.05
Benzo (b) fluorene	0.814
C29-Hopane	10.0
18a-Oleanane	10.0
C30-Hopane	10.0
C20-TAS	10.0
C21-TAS	10.0
C26(20S)-TAS	10.0
C26(20R)/C27(20S)-TAS	10.0
C28(20S)-TAS	10.0
C27(20R)-TAS	10.0
C28(20R)-TAS	10.0

Table B4. Pesticide method detection limits. Sample size = 2.17g.

OCs	Tissue MDLs (ng/dry g)
Aldrin	0.22
Dieldrin	0.24
Endrin	0.38
Heptachlor	0.23
Heptachlor-Epoxide	0.24
Oxychlordane	0.21
Alpha-Chlordane	0.25
Gamma-Chlordane	0.24
Trans-Nonachlor	0.24
Cis-Nonachlor	0.21
Alpha-HCH	0.25
Beta-HCH	0.27
Delta-HCH	0.23
Gamma-HCH	0.26
DDMU	0.28
2,4'-DDD	0.25
4,4'-DDD	0.24
2,4'-DDE	0.24
4,4'-DDE	0.28
2,4'-DDT	0.26

OCs	Tissue MDLs (ng/dry g)
4,4'-DDT	0.22
1,2,3,4-Tetrachlorobenzene	0.26
1,2,4,5-Tetrachlorobenzene	0.24
Hexachlorobenzene	0.29
Pentachloroanisole	0.25
Pentachlorobenzene	0.23
Endosulfan II	0.25
Endosulfan I	0.26
Endosulfan Sulfate	0.26
Mirex	0.22
Chlorpyrifos	0.34
PCB1	0.43
PCB7/9	0.43
PCB8/5	0.43
PCB15	0.43
PCB16/32	0.40
PCB18	0.40
PCB22/51	0.40
PCB24/27	0.40
PCB25	0.40
PCB26	0.40
PCB28	0.43
PCB29	0.29
PCB31	0.40
PCB33/53/20	0.40
PCB40	0.39
PCB41/64	0.39
PCB42/59/37	0.39
PCB43	0.39
PCB44	0.39
PCB45	0.39
PCB46	0.39
PCB47/48/75	0.39
PCB49	0.39
PCB52	0.39
PCB56/60	0.39
PCB66	0.31
PCB70	0.39
PCB74/61	0.39

OCs	Tissue MDLs (ng/dry g)
PCB77	0.39
PCB81	0.39
PCB82	0.42
PCB83	0.42
PCB84	0.42
PCB85	0.42
PCB86	0.42
PCB87/115	0.31
PCB88	0.42
PCB92	0.42
PCB95	0.42
PCB97	0.42
PCB99	0.42
PCB101/90	0.42
PCB105	0.35
PCB107	0.42
PCB110/77	0.28
PCB114/131/122	0.42
PCB118	0.33
PCB126	0.42
PCB128	0.43
PCB129/126	0.60
PCB136	0.60
PCB138/160	0.60
PCB141/179	0.60
PCB146	0.60
PCB149/123	0.60
PCB151	0.60
PCB153/132	0.31
PCB156/171/202	0.60
PCB158	0.60
PCB166	0.60
PCB167	0.60
PCB169	0.60
PCB170/190	0.37
PCB172	0.29
PCB174	0.29
PCB176/137	0.29
PCB177	0.29

OCs	Tissue MDLs (ng/dry g)
PCB178	0.29
PCB180	0.29
PCB183	0.29
PCB185	0.29
PCB187	0.21
PCB189	0.29
PCB191	0.29
PCB194	0.30
PCB195/208	0.30
PCB196/203	0.30
PCB199	0.50
PCB200	0.30
PCB201/157/173	0.30
PCB205	0.30
PCB206	0.35
PCB209	0.30

Table B5. Butyltin reporting limits. Sample size = 1.81g.

BTs	Tissue RLs (ng/Sn dry g)
Monobutyltin	1.10
Dibutyltin	1.10
Tributyltin	1.10
Tetrabutyltin	1.10

Table B6. PBDE method detection limits. Sample size = 2.02g.

PBDE	Tissue (ng/dry g)
PBDE 1 (2-MonoBDE)	0.389
PBDE 2 (3-MonoBDE)	0.389
PBDE 3 (4-MonoBDE)	0.389
PBDE 10 (2,6-DiBDE)	0.389
PBDE 7 (2,4-DiBDE)	0.389
PBDE 11 (3,3'-DiBDE)	0.389
PBDE 8 (2,4'-DiBDE)	0.389
PBDE 12 (3,4-DiBDE)	0.389
PBDE 13 (3,4'-DiBDE)	0.389
PBDE 15 (4,4'-DiBDE)	0.389
PBDE 32 (2,4',6-TriBDE)	0.389
PBDE 30 (2,4,6-TriBDE)	0.389
PBDE 17 (2,2',4-TriBDE)	0.389
PBDE 25 (2,3',4-TriBDE)	0.389
PBDE 33 (2',3,4-TriBDE)	0.189
PBDE 28 (2,4,4'-TriBDE)	0.419
PBDE 35 (3,3',4-TriBDE)	0.389
PBDE 37 (3,4,4'-TriBDE)	0.389
PBDE 75 (2,4,4',6-TetraBDE)	0.456
PBDE 49/71 (2,2',4,5'-TetraBDE/2,3',4',6-PTetraBDE)	0.333
PBDE 47 (2,2',4,4'-TetraBDE)	0.456
PBDE 66 (2,3',4,4'-TetraBDE)	0.250
PBDE 77 (3,3',4,4'-TetraBDE)	0.456
PBDE 100 (2,2',4,4',6-PentaBDE)	0.810
PBDE 119 (2,3',4,4',6-PentaBDE)	0.416
PBDE 99 (2,2',4,4',5-PentaBDE)	0.416
PBDE 116 (2,3,4,5,6-PentaBDE)	0.416
PBDE 118 (2,3',4,4',5-PentaBDE)	0.416
PBDE 126 (3,3',4,4',5-PentaBDE)	0.416
PBDE 85 (2,2',3,4,4'-PentaBDE)	0.854
PBDE 155 (2,2',4,4',6,6'-HexaBDE)	0.310
PBDE 154 (2,2',4,4',5,6'-HexaBDE)	0.385
PBDE 153 (2,2',4,4',5,5'-HexaBDE)	0.336
PBDE 138 (2,2',3,4,4',5'-HexaBDE)	0.310
PBDE 166 (2,3,4,4',5,6-HexaBDE)	0.310
PBDE 183 (2,2',3,4,4',5',6-HeptaBDE)	0.285
PBDE 181 (2,2',3,4,4',5,6-HeptaBDE)	0.285
PBDE 190 (2,3,3',4,4',5,6-HeptaBDE)	0.216

PBDE	Tissue (ng/dry g)
PBDE-202 (2,2',3,3',5,5',6,6'-OctaBDE)	0.216
PBDE-201 (2,2',3,3',4,5',6,6'-OctaBDE)	0.216
PBDE-204 (2,2',3,4,4',5,6,6'-OctaBDE)	0.216
PBDE-197 (2,2',3,3',4,4',6,6'-OctaBDE)	0.216
PBDE-198/199/203/200 (OctaBDE)	0.216
PBDE-196 (2,2',3,3',4,4',5,6'-OctaBDE)	0.216
PBDE-205 (2,3,3',4,4',5,5',6-OctaBDE)	0.216
PBDE-194 (2,2',3,3',4,4',5,5'-OctaBDE)	0.216
PBDE-195 (2,2',3,3',4,4',5,6-OctaBDE)	0.216
PBDE-208 (2,2',3,3',4,5,5',6,6-NonaBDE)	0.216
PBDE-207 (2,2',3,3',4,4',5,6,6'-NonaBDE)	0.216
PBDE-206 (2,2',3,3',4,4',5,5',6-NonaBDE)	0.216
PBDE-209 (DecaBDE)	3.23

Table B7. PBB method detection limits. Sample size = 2.02g.

PBB	Tissue (ng/dry g)
PBB 1 (2-MonoBB)	1.76
PBB 2 (3-MonoBB)	1.56
PBB 3 (4-MonoBB)	1.78
PBB 4 (2,2'-DiBB)	1.47
PBB 10 (2,6-DiBB)	1.79
PBB 7 (2,4-DiBB)	1.91
PBB 9 (2,5-DiBB)	1.85
PBB 15 (4,4'-DiBB)	1.94
PBB 30 (2,4,6-TriBB)	1.84
PBB 18 (2,2',5-TriBB)	1.57
PBB 26 (2,3',5-TriBB)	1.71
PBB 31 (2,4',5-TriBB)	1.98
PBB 53 (2,2',5,6'-TetraBB)	2.32
PBB 52 (2,2',5,5'-TetraBB)	2.39
PBB 49 (2,2',4,5'-TetraBB)	2.01
PBB 103 (2,2',4,5',6-PentaBB)	1.07
PBB 80 (3,3',5,5'-TetraBB)	1.62
PBB 77 (3,3',4,4'-TetraBB)	2.44
PBB 155 (2,2',4,4',6,6'-HexaBB)	1.82

Table B8. Method performance criteria for trace metals.

Sample Type	Minimum Frequency	Acceptance Criteria
Method Blank	One per batch/every 20 samples	No analytes exceed 2X the method detection limits. Higher blank levels are acceptable as long as the observed sample concentrations are not significantly impacted by blank concentrations
Matrix Spike Blank Spike	One per batch/every 20 samples	The recovery for all analytes is between 80-120% for valid spikes. As long as the spiking level is "valid", or at least as high as the concentration of the analyte in the unspiked sample
Laboratory Duplicate	One per batch/every 20 samples	The QA criterion for valid duplicates and spiked duplicates is $\pm 30\%$. Where concentrations are greater than 3x the MDL
Reference Materials NIST SRM	One per batch/every 20 samples	$\pm 20\%$ the certified limit. Where concentrations are greater than 3x the MDL

Table B9. Method performance criteria for extended PAH (parent and alkyl homologs) and related compounds.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Tuning	Prior to every sequence	Tune as specified in laboratory SOP	Resolve before proceeding.
Initial Calibration (All parent PAH and selected alkyl homologue PAH)	Prior to every sequence, or as needed based on continuing calibration/verification check.	6-point calibration curve over two orders of magnitude RSD $\leq 20\%$	Resolve before proceeding.
Continuing Calibration Verification (CCV)	Every 12 hours or 6-9 field samples	%D $\leq 25\%$, No more than 2 analytes can be between 25% and 35%	Perform instrument maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCV is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
SRM 1941b for sediment; SRM 1974c for tissue If available use SRMs for appropriate matrices	One per batch/every 20 field samples	Within $\pm 30\%$ of NIST 95% uncertainty range for analytes within the quantitation range. No more than 2 analytes may exceed this criterion.	Resolve before proceeding.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
SRM 2779 Reference Oil	One per batch/every 20 field samples	Peak resolution >80% of 4/9-methylphenanthrene from 1-methylphenanthrene (m/z 192). Within $\pm 20\%$ of NIST 95% uncertainty range for analytes within the quantitation range. No more than 2 analytes may exceed this criterion.	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments, Soils, Tissues only)	One per batch/every 20 field samples	%R 40% - 120% for target analytes, except biphenyl (40%-140%), decalin (25%-120%) and perylene (10-120%); RPD $\leq 30\%$, average %R 60-120% for valid spikes. No more than 2 analytes may exceed 40-120% recovery or >35% RPD.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Blank Spike/Blank Spike Duplicate	One per batch/every 20 field samples	See MS/MSD criteria above.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.

Table B10. Method performance criteria for extended PAH (parent and alkyl homologs) and related compounds.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Method Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 3x target MDL unless analyte not detected in associated sample(s) or analyte concentration >10x blank value	Resolve before proceeding. Lab manager may be contacted to resolve issues surrounding 'minor exceedance'.
Laboratory Duplicate (not required for aqueous samples)	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is 3x greater than the MDL, no more than 2 individual analyte RPDs with conc. 3x MDL can exceed 35%.	Evaluate impact to data, discuss with lab manager, and determine if corrective action is needed.
Mass Discrimination	Initial calibration and CCVs (mid-level)	Ratio for the concentration of Benzo [g,h,i] perylene to phenanthrene $\geq$ 0.70	Resolve before proceeding.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in the associated calibration standard	Resolve before proceeding.
Surrogates	Every sample	%R 40-120% except d12-perylene which is 10-120%	Re-extract affected samples. Evaluate impact to data, discuss with lab manager, if corrective action is needed.

Table B11. Method performance criteria for chlorinated hydrocarbon compounds.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Initial Calibration or Interspersed Calibration	Prior to every sequence, or as needed based on continuing calibration/verification check/ Interspersed calibration analyzed with every sequence	Minimum of 5-point calibration curve over two orders of magnitude Calibration correlation coefficient >.997	Resolve before proceeding.
Continuing Calibration Verification (CCV)	Every 12 hours or every 6-8 field samples	RPD $\leq$ 25% for 90% of analytes RPD $\leq$ 35% for 10% of analytes	Perform instrument maintenance. Re-analyze affected samples.
Initial Calibration Verification (Second Source or can be met if CCV is second source)	Per initial calibration	%R target analytes 80-120%	Resolve before proceeding.
SRM 1941b for sediment; SRM 1974c for tissue If available use SRMs for appropriate matrices	One per batch/every 20 field samples	Within $\pm$ 30% of NIST 95% uncertainty range for analytes within the quantitation range. No more than 2 analytes may exceed this criterion.	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments, Soils, Tissues only)	One per batch/every 20 field samples	%R 40% - 120% for target analytes, except Chlorpyrifos and Endosulfan II, RPD $\leq$ 30%, average %R 60-120% for valid spikes. No more than 2 analytes may exceed 40-120% recovery or >35% RPD.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Blank Spike/Blank Spike Duplicate	One per batch/every 20 field samples	See MS/MSD criteria above.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Method Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 3x target MDL unless analyte not detected in associated samples(s) or analyte concentration >10x blank value	Resolve before proceeding. Lab manager may be contacted to resolve issues surrounding 'minor exceedance.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Laboratory Duplicate (not required for aqueous samples)	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is 10x greater than the MDL, no more than 2 individual analyte RPDs with conc. 10x MDL can exceed 35%.	Evaluate impact to data, discuss with lab manager, and determine if corrective action is needed.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in a mid-level initial calibration standard	Resolve before proceeding.
Surrogates	Every sample	%R 50-150%	Re-extract affected samples. Evaluate impact to data, discuss with lab manager, if corrective action is needed.

Table B12. Method performance criteria for butyltins.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Tuning	Prior to every sequence	Tune as specified in laboratory SOP	Resolve before proceeding.
Initial Calibration	Prior to every sequence, or as needed based on continuing calibration verification.	6-point linear calibration curve over two orders of magnitude Calibration correlation coefficient $>.997$	Resolve before proceeding.
Continuing Calibration Verification (CCV)	Every 12 hours or 10-12 injections	RPD $\leq$ 25%	Perform instrument maintenance. Re-analyze affected samples.
PACs-2 for sediment LCS for tissue If available use SRMs for appropriate matrices	One per batch/every 20 field samples	Within $\pm 30\%$ of NIST 95% uncertainty range for analytes within the quantitation range. No more than 1 analyte may exceed this criterion.	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments, Soils, Tissues only)	One per batch/every 20 field samples	%R 40% - 120% for target analytes, except monobutyltin (10%-120%); RPD $\leq 30\%$, No more than 1 analyte may exceed 40-120% recovery or $>35\%$ RPD.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Blank Spike/Blank Spike Duplicate	One per batch/every 20 field samples	See MS/MSD criteria above.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Method Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 3x target MDL unless analyte not detected in associated sample(s) or analyte concentration >10x blank value	Resolve before proceeding. Lab manager may be contacted to resolve issues surrounding 'minor exceedance'.
Laboratory Duplicate (not required for aqueous samples)	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is 10x greater than the MDL, no more than 1 individual analyte RPD with conc. 10x MDL can exceed 35%.	Evaluate impact to data, discuss with lab manager, and determine if corrective action is needed.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in a mid-level initial calibration standard	Resolve before proceeding.
Surrogates	Every sample	%R 50-150%	Re-extract affected samples. Evaluate impact to data, discuss with lab manager, if corrective action is needed.

Table B13. Method performance criteria for polybrominated diphenyl ether and polybrominated biphenyl compounds.

Element or Sample Type	Minimum Frequency	Measurement Quality Objective/Acceptance Criteria	Corrective Action
Tuning	Prior to every sequence	Tune as specified in laboratory SOP	Resolve before proceeding.
Initial Calibration	Prior to every sequence, or as needed based on continuing calibration/verification check.	5-point calibration curve over two orders of magnitude RSD $\leq$ 20%	Resolve before proceeding.
Continuing Calibration Verification (CCV)	Every 12 hours or 6-10 injections	%D $\leq$ 25%, No more than 2 analytes can be between 25% and 35% %D. Average %D $\leq$ 15	Perform instrument maintenance. Re-analyze affected samples.
SRM 1941b for sediment; SRM 1974c for tissue If available use SRMs for appropriate matrices	One per batch/every 20 field samples	Within $\pm$ 30% of NIST 95% uncertainty range for analytes within the quantitation range. No more than 2 analytes may exceed this criterion.	Resolve before proceeding.
Matrix Spike/Matrix Spike Duplicate (Sediments, Soils, Tissues only)	One per batch/every 20 field samples	%R 40% - 120% for target analytes; RPD $\leq$ 30%, average %R 60-120% for valid spikes. No more than 2 analytes may exceed 40-120% recovery or >35% RPD.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Blank Spike/Blank Spike Duplicate	One per batch/every 20 field samples	See MS/MSD criteria above.	Evaluate impact to data, discuss with lab manager, determine if corrective action is needed.
Method Blank	One per batch/every 20 field samples	No more than 2 analytes to exceed 3x target MDL unless analyte not detected in associated sample(s) or analyte concentration >10x blank value	Resolve before proceeding. Lab manager may be contacted to resolve issues surrounding 'minor exceedance'.
Laboratory Duplicate (not required for aqueous samples)	One per batch/every 20 field samples	RPD $\leq$ 30% if analyte concentration is 3x greater than the MDL, no more than 2 individual analyte RPDs with conc. 3x MDL can exceed 35%.	Evaluate impact to data, discuss with lab manager, and determine if corrective action is needed.
Internal Standard (IS)	Every sample	50% - 200% of the area of the IS in the associated calibration standard	Resolve before proceeding.
Surrogates	Every sample	%R 50-150%	Re-extract affected samples. Evaluate impact to data, discuss with lab manager, if corrective action is needed.

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