ANALYTICAL QUALITY ASSURANCE PLAN

HUDSON RIVER NATURAL RESOURCE DAMAGE ASSESSMENT

HUDSON RIVER NATURAL RESOURCE TRUSTEES

STATE OF NEW YORK

U.S. DEPARTMENT OF COMMERCE

U.S. DEPARTMENT OF THE INTERIOR

FINAL
PUBLIC RELEASE VERSION*

VERSION 2.0

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**Names of certain individuals and affiliations have been removed to maintain confidentiality*

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Approved for Release:

Project Coordinator

Quality Assurance Coordinator

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INTRODUCTION

The Hudson River Natural Resource Trustees (Trustees) are conducting a natural resource damage assessment (NRDA) of the Hudson River. The Trustees are the State of New York acting through the New York State Department of Environmental Conservation (NYSDEC), the Department of the Interior acting through the U.S. Fish and Wildlife Service (USFWS), and the Department of Commerce acting through the National Oceanic and Atmospheric Administration (NOAA). This NRDA involves analysis of soil, sediment, fish, and wildlife samples collected from the Hudson River and surrounding area. The primary analytes of concern are polychlorinated biphenyl (PCB) congeners, PCB homologues, and Total PCBs. In addition, samples may be analyzed for the following parameters: coplanar PCB congeners, dioxin/furans, organochlorine pesticides, PBDEs and metals. The Trustees also plan to conduct analytical tests such as moisture, total extractable organics (TEO)[1], total organic carbon (TOC), and grain size to support data interpretation.

This Analytical Quality Assurance (QA) Plan describes the minimum requirements for the chemical analysis of the environmental samples that are collected in support of the NRDA. This plan does not address the actual field collection or QA measures required in the field during generation of these samples. The requirements specified in this plan are designed to: (1) monitor the performance of the measurement systems to maintain statistical control and provide rapid feedback so that corrective measures can be taken before data quality is compromised and; (2) verify that reported data are sufficiently complete, comparable, representative, unbiased and precise so as to be suitable for their intended use.

This Analytical QA Plan is consistent with the intent of NRDA regulations, as provided in *43 CFR Subtitle A, subpart C* and satisfies the requirements listed in the relevant EPA guidance for QA plans (USEPA 2001 and USEPA 1998) as far as the documents relate to analytical testing services. This Analytical QA plan will be revised as appropriate, as changes are made to the damage assessment and the QA program. This Analytical QA Plan supercedes earlier versions of the Hudson River NRDA Analytical QA Plans, including version 1.0 (Hudson River Natural Resource Trustees 2002).

[1] Total extractable organics (TEO) is also commonly referred to as "lipids". For the purposes of this project the term "TEO" will be used rather than "lipids" because the percent TEO represents the total quantity of material solvent-extracted from a sample prior to organic analyses. This solvent-extract may contain additional components other than lipids.

1.0 PROJECT DESCRIPTION

The primary analytes of interest are PCBs. This includes quantitation of specific individual PCB congeners by low resolution mass spectrometry (LRMS), calculation of concentrations for each homologue group, and summing of the homologue groups for a Total PCB value. The congeners of potential interest and target detection limits are listed in **Table 1.1a**.

Samples may also be analyzed for PCB coplanar and mono-ortho substituted PCB congeners by high resolution mass spectrometry (HRMS) (**Tables 1.1b & 1.1c**), dioxin/furans by HRMS (**Table 1.1d**), PBDEs (**Table 1.1e**), organochlorine pesticides (**Table 1.1f**) and metals (**Table 1.1g**). Analytes and target detection (organics) or quantitation (metals) limits are listed in the referenced tables.

For organic analyses, detection limits[2] are based on a minimum sample size of 3 grams. Target detection limits may be raised proportionally for reduced sample size. If a laboratory method detection limit does not meet the target detection limit, a higher limit is acceptable if approved by the QA Coordinator. For metals analyses, quantitation[3] limits are based on a minimum sample size of 1 gram.

Additional analyses to support the Total PCB and PCB congener investigations include percent moisture (tissue and soil/sediment), percent TEO (tissue), total organic carbon (soil/sediment) and grain size (soil/sediment). Reporting limits for these analyses are provided in **Table 1.1h**.

Matrices for analysis will include a wide range of tissues (*e.g.*, vegetation, fish, birds, mammals), soil, and/or sediment. The work plans and associated QA plans under which these samples were generated or collected are independent documents and not included or considered herein. This Analytical QA Plan describes the minimum requirements to be taken to provide for the chemical analyses (and associated physical normalizing parameters, i.e. total extractable organics, percent moisture, total organic carbon, grain size) of the previously generated or collected samples in a technically sound and legally defensible manner.

[2] The method detection limit is the lowest concentration that can be detected by an instrument with correction for the effects of sample matrix and method-specific parameters such as sample preparation.

[3] The target quantitation limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy (i.e., the DQOs) during routine laboratory operating conditions.

TABLE 1.1a

PCB Congener Target Compound List

BZ# 8	BZ# 74	BZ# 126[1]	BZ# 170
BZ# 18	BZ# 77[1]	BZ# 128	BZ# 174
BZ# 28	BZ# 81[1]	BZ# 138	BZ# 177
BZ# 31	BZ# 87	BZ# 146	BZ# 180
BZ# 44	BZ# 95	BZ# 149	BZ# 183
BZ# 45	BZ# 99	BZ# 151	BZ# 187
BZ# 47	BZ# 101	BZ# 153	BZ# 189
BZ# 49	BZ# 105	BZ# 156	BZ# 194
BZ# 52	BZ# 110	BZ# 157	BZ# 195
BZ# 56	BZ# 114	BZ# 158	BZ# 201
BZ# 66	BZ# 118	BZ# 167	BZ# 206
BZ# 70	BZ# 123[1]	BZ# 169[1]	BZ# 209
Total Monochlorobiphenyls		Total Dichlorobiphenyls	
Total Trichlorobiphenyls		Total Tetrachlorobiphenyls	
Total Pentachlorobiphenyls		Total Hexachlorobiphenyls	
Total Heptachlorobiphenyls		Total Octachlorobiphenyls	
Total Nonachlorobiphenyls		Decachlorobiphenyl	
Total PCBs			

PCB Congener Target Detection Limits (LRMS):

Soil/sediment = 0.1 ng/g dry weight

Tissue = 0.1 ng/g wet weight

[1] These concentrations should be interpreted as maximum values when obtained from LRMS analysis due to the potential for interferences.

PCB Homologue and Total PCB Target Detection Limits (LRMS):

Soil/sediment = 10 ng/g dry weight

Tissue = 10 ng/g wet weight

TABLE 1.1b

PCB Coplanar Target Compound List

BZ# 77
BZ# 81
BZ# 126
BZ# 169

PCB Coplanar Target Detection Limits (HRMS):

Soil/sediment = 0.001 ng/g dry weight

Tissue = 0.001 ng/g wet weight

TABLE 1.1c

PCB Coplanar & Mono-ortho Congener Target Compound List

BZ# 77	BZ# 118	BZ# 157
BZ# 81	BZ# 123	BZ# 167
BZ# 105	BZ# 126	BZ# 169
BZ# 114	BZ# 156	BZ# 189

PCB Coplanar & Mono-ortho Congener Target Detection Limits (HRMS):

Soil/sediment = 0.001 ng/g dry weight

Tissue = 0.001 ng/g wet weight

TABLE 1.1d

Dioxin and Furan Target Compound List

2,3,7,8-TCDD	2,3,7,8-TCDF
1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDF
1,2,3,4,7,8-HxCDD	2,3,4,7,8-PeCDF
1,2,3,6,7,8-HxCDD	1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDD	1,2,3,7,8,9-HxCDF
OCDD	2,3,4,6,7,8-HxCDF
Total Tetra-Dioxins (TCDD)	1,2,3,4,6,7,8-HpCDF
Total Penta-Dioxins (PeCDD)	1,2,3,4,7,8,9-HpCDF
Total Hexa-Dioxins (HxCDD)	OCDF
Total Hepta-Dioxins (HpCDD)	Total Tetra-Furans (TCDF)
	Total Penta-Furans (PeCDF)
	Total Hexa-Furans (HxCDF)
	Total Hepta-Furans (HpCDF)

Dioxin and Furan Target Detection Limits (HRMS):

Soil/sediment = 0.0005 ng/g dry weight

Tissue = 0.0005 ng/g wet weight

EXCEPT OCDD & OCDF at 0.0025 ng/g

TABLE 1.1e
Polybrominated Diphenyl Ether (PBDE) Target Compound List

2,2',4-TriBDE (17)	2,3',4,4',6-PentaBDE (119)
2,3',4-TriBDE (25)	3,3',4,4',5-PentaBDE (126)
2,4,4'-TriBDE (28)	2,2',3,4,4',5-HexaBDE (138)
2,4',6-TriBDE (30)	2,2',3,4,4',6-HexaBDE (140)
2',3,4-TriBDE (33)	2,2',4,4',5,5'-HexaBDE (153)
3,3',4-TriBDE (35)	2,2',4,4',5,6'-HexaBDE (154)
3,4,4-TriBDE (37)	2,2',4,4',6,6'-HexaBDE (155)
2,2',4,4'-TetraBDE (47)	2,3,4,4',5,6-HexaBDE (166)
2,2',4,5'-TetraBDE (49)	2,2',3,4,4',5,6-HeptaBDE (181)
2,3',4,4'-TetraBDE (66)	2,2',3,4,4',5',6-HeptaBDE (183)
2,3',4',6-TetraBDE (71)	2,3,3',4,4',5,6-HeptaBDE (190)
3,3',4,4'-TetraBDE (77)	2,2',3,4,4',5,5',6-OctaBDE (203)
2,2',3,4,4'-PentaBDE (85)	2,2',3,3',4,4',5,5',6-NonaBDE (206)
2,2',4,4',5-PentaBDE (99)	2,2',3,3',4,4',5,6,6'-NonaBDE (207)
2,2',4,4',6-PentaBDE (100)	2,2',3,3',4,5,5',6,6'-NonaBDE (208)
2,3,3',4,4'-PentaBDE (105)	2,2',3,3',4,4',5,5',6,6'-DecaBDE (209)
2,3,4,5,6-PentaBDE (116)	

PBDE Target Detection Limits (HRMS):

Soil/sediment = 0.005 ng/g wet weight,
EXCEPT BDEs 47, 99 & NonaBDEs at 0.02 ng/g; DecaBDE at 1 ng/g
Tissue = 0.005 ng/g wet weight,
EXCEPT BDEs 47, 99 & NonaBDEs at 0.02 ng/g; DecaBDE at 1 ng/g

TABLE 1.1f

Organochlorine Pesticide Target Compound List

Aldrin	Endosulfan I
Alpha-BHC	Endosulfan II
Beta-BHC	Endosulfan sulfate
Gamma-BHC	Endrin
Alpha-Chlordane	Endrin Aldehyde
Gamma-Chlordane	Endrin Ketone
Chlordane	Heptachlor
2,4'-DDD	Heptachlor Epoxide
2,4'-DDE	Hexachlorobenzene
2,4'-DDT	Methoxychlor
4,4'-DDD	cis-Nonachlor
4,4'-DDE	trans-Nonachlor
4,4'-DDT	Oxychlordane
Dieldrin	Toxaphene

Organochlorine Pesticide Target Detection Limits (GC-ECD or LRMS):

Soil/sediment = 1.0 ng/g dry weight

EXCEPT Aldrin, Heptachlor, Methoxychlor, Oxychlordane at 2.0 ng/g; Toxaphene at 5.0 ng/g

Tissue = 1.0 ng/g wet weight

EXCEPT Aldrin, Heptachlor, Methoxychlor, Oxychlordane at 2.0 ng/g; Toxaphene at 5.0 ng/g

TABLE 1.1g
Metals Target Compound List

Analyte	Method	Tissue Target Quantitation Limit[1] (mg/kg)	Sediment Target Quantitation Limit[1] (mg/kg)
Aluminum	ICP/ICP-MS	2	2
Antimony	ICP/ICP-MS	0.05	0.05
Arsenic	ICP/ICP-MS	0.02	0.01
Barium	ICP/ICP-MS	0.2	0.05
Beryllium	ICP/ICP-MS	0.02	0.01
Cadmium	ICP/ICP-MS	0.01	0.01
Calcium	ICP/ICP-MS	50	10
Chromium	ICP/ICP-MS	0.02	0.05
Cobalt	ICP/ICP-MS	0.01	0.01
Copper	ICP/ICP-MS	0.05	0.05
Iron	ICP/ICP-MS	2.0	5.0
Lead	ICP/ICP-MS	0.02	0.05
Magnesium	ICP/ICP-MS	5.0	1.0
Manganese	ICP/ICP-MS	0.2	1.0
Mercury	CVAA	0.02	0.02
Nickel	ICP/ICP-MS	0.05	0.05
Potassium	ICP/ICP-MS	25	5.0
Selenium	ICP/ICP-MS	0.05	0.05
Silver	ICP/ICP-MS	0.02	0.02
Sodium	ICP/ICP-MS	200	5.0
Thallium	ICP/ICP-MS	0.01	0.01
Vanadium	ICP/ICP-MS	0.05	0.05
Zinc	ICP/ICP-MS	0.2	0.5

[1] Target quantitation limits are based on 50 ml final volume, 1g mass digested and diluted 1:2 at the instrument prior to analysis. Tissues are reported on a wet weight basis, sediments are reported on a dry weight basis.

TABLE 1.1h
Supporting Analyses Target Reporting Limits

Analyte	Soil/Sediment Reporting Limit(%)	Tissue Reporting Limit(%)
TEO	NA	0.01
Percent Moisture	0.01	0.01
TOC (2 Replicates)	0.01	NA
Grain Size (5 Fractions)	0.01	NA

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 ASSESSMENT MANAGERS

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The Assessment Managers are the designated representatives (from NOAA, DOI, and NYSDEC) who are responsible for the review and acceptance of specific work plans and associated QA plans.

2.2 PROJECT COORDINATOR

The Project Coordinator is responsible for administration of the laboratory(ies) contract(s). The Project Coordinator also remains informed of the identification of Principal Investigators and staffing for each of the studies to be conducted for the NRDA. The Principal Investigators for each study will be the end-users of the data produced under the Analytical QA Plan. The Project Coordinator will oversee the proper scheduling and transmittal of the data from the time of sampling to data reporting.

2.3 QUALITY ASSURANCE COORDINATOR

The QA Coordinator reports directly to the Assessment Managers. The QA Coordinator is responsible for the implementation of this Analytical QA Plan, as described in Appendix A (QA Assurance Management) of the *Hudson River NRDA Plan*. The QA Coordinator will receive assistance in the coordination and performance of laboratory technical audits and independent data validation from the QA Contractor. The QA Coordinator has the authority and responsibility to cease or temporarily halt activities not in keeping with this QA Plan. The QA Coordinator will work closely with laboratory representatives and the project team to assure that project and data quality objectives are met.

2.4 ANALYTICAL LABORATORIES

The primary contractors selected by the Trustees for analytical work in support of the Hudson River NRDA are Alpha Woods Hole Lab (AWHL), Raynham, Massachusetts, and Axys Analytical Services, Limited (Axys), Sidney, British Columbia. The laboratory project managers are responsible for assuring that all analyses performed meet project and data quality objectives. The Laboratory Project Managers are:

Peter Kane
Alpha Woods Hole Lab
375 Paramount Drive, Suite 2
Raynham, MA 02767-5154
(508)822-9300 FAX (508)822-3288
pkane@alphalab.com

Pam Riley
Axys Analytical Services, Limited
2045 Mills Road West
Sidney, British Columbia, Canada V8L358
(250)655-5800 FAX (250)655-5811
priley@axys.com

3.0 SAMPLE HANDLING AND CHAIN OF CUSTODY PROCEDURES

Chain of custody procedures will be used for all samples throughout the analytical process and for all data and data documentation, whether in hard copy or electronic format. Sampling procedures, including sample collection and documentation, are part of the work plans of the individual projects and as such, are not considered here.

3.1 SAMPLE PRESERVATION

Tissue, sediment and soil samples will be collected for analysis. Sample preservation and field treatment of samples for analyses should be described in relevant field work plans. Briefly, tissue, sediment and soil samples for chemical analysis should be sealed in appropriate containers and frozen as soon after collection as possible. The samples should be maintained at -20°C until prepared for analysis.

3.2 SAMPLE HOLDING TIMES

The primary analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage (Wise et al. 1989). Thus, we are not establishing a maximum holding time for samples. Percent solids or moisture will be reported with each tissue, sediment or soil result to allow for normalization if there are changes in sample moisture content during sample storage.

3.3 CHAIN OF CUSTODY

Each container is considered to be an individual sample and will be assigned a unique identification number and have a separate entry on the chain of custody record.

Chain of custody records will be completed in ink.

A sample is considered in "custody" if:

- it is in the custodian's actual possession or view, or
- it is retained in a secured place (under lock) with restricted access, or
- it is placed in a container and secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s).

Samples are kept in the custody of designated sampling and/or field personnel until shipment.

3.4 SAMPLE SHIPPING

Any transfer or movement of samples will use chain of custody procedures. The original signed and dated chain of custody record accompanies the sample(s); a copy is retained by the sample shipper. All shipments will comply with DOT regulations (*49CFR, Parts 172 and 173*).

3.5 SAMPLE RECEIPT

Immediately upon receipt of samples, the recipient will review the shipment for consistency with the accompanying chain of custody record and sample condition before signing and dating the chain of custody record. Sample condition(s) will be noted on the original chain of custody sheet at this time. If there are any discrepancies between the chain of custody record and the sample shipment, the recipient will contact the sample shipper immediately in an attempt to reconcile these differences. Resolution of any discrepancies will accompany the data report.

3.6 INTRA-LABORATORY SAMPLE TRANSFER

The laboratory sample custodian or designee will maintain a laboratory sample-tracking record, similar to the chain of custody record, that will follow each sample through all stages of laboratory processing. The sample-tracking record will show the name or initials of responsible individuals, date of sample extraction or preparation, and sample analysis.

3.7 INTER-LABORATORY SAMPLE TRANSFER

Transfer of samples from one analytical laboratory to another, e.g. for grain size or TOC analysis, will follow chain of custody, sample shipping and receipt procedures described above.

3.8 SAMPLE ARCHIVAL

All unanalyzed samples and unutilized sample aliquots or extracts will be held by the laboratory in a manner to preserve sample integrity at a secure location with chain of custody procedures for one (1) year after the QA Contractor has validated the data package for that particular set of samples. All archived materials will be accessible for review upon request. At the end of the archival period, the laboratory shall contact the QA Coordinator to obtain directions for handling remaining samples. The samples will not be disposed of by the laboratory unless provided with written approval from the Assessment Manager.

3.9 DATA AND DATA DOCUMENTATION

All data and data documentation, whether in hard copy or electronic format, are the responsibility of the QA Coordinator acting on behalf of Hudson River Case Management Team. The laboratory case narrative and any summary information will be clearly marked with "Privileged and Confidential, FOIA/FOIL Exempt, Not for Release."

The QA Contractor will receive from the laboratories data tables and QA documentation suitable for QA assessment/data validation. A copy of the data and data documentation developed by the laboratory for a given data package will be kept by the laboratory in a secure location under chain of custody procedures for five (5) years after the QA Contractor has validated that data package. All archived materials will be accessible for review upon request. These materials will become the responsibility of the Assessment Manager upon termination of the laboratory archival period.

The original data will be transferred from the laboratory to the QA Contractor by means such that a signature is required at the time of document delivery. The QA Contractor will document receipt of packages and maintain a record of the method and date of data submittal with the complete data package. The QA Contractor will maintain the copy of the data packages and related validation documentation in a secure location for a period of one (1) year from the date of validation. These materials will become the responsibility of the Assessment Manager upon termination of the QA archival period.

4.0 LABORATORY OPERATIONS

All laboratories providing analytical support for the Hudson River Damage Assessment must have the appropriate facilities to store and prepare samples, and appropriate instrumentation and staff to provide data of the required quality within the time period dictated. Laboratories are expected to conduct operations using good laboratory practices, including:

- Training and appropriate certification of personnel.
- A program of scheduled maintenance of analytical balances, laboratory equipment and instrumentation.
- Routine checking of analytical balances using a set of standard reference weights (ASTM class, NIST Class S-1, or equivalents).
- Recording all analytical data in logbooks; each entry signed and dated by the analyst.
- Monitoring and documenting the temperatures of cold storage areas and freezer units.

Laboratory operations will be evaluated by the QA Coordinator through technical systems audits, performance evaluation studies, and performance in the NIST-managed intercomparison program (PCBs only). Personnel in any laboratory performing analyses for this damage assessment should be well versed in good laboratory practices, including standard safety procedures. It is the responsibility of the laboratory manager and /or supervisor to ensure that safety training is mandatory for all laboratory personnel. The laboratory is responsible for maintaining a current safety manual in compliance with the Occupational Safety and Health Administration (OSHA) or equivalent state or local regulations. Proper procedures for safe storage, handling and disposal of chemicals should be followed at all times; each chemical should be treated as a potential health hazard and good laboratory practices should be implemented accordingly.

4.1 QUALITY ASSURANCE DOCUMENTATION

All laboratories must have the latest revision of the Hudson River NRDA Analytical QA Plan. In addition, the following documents and information must be current and available to all laboratory personnel participating in the processing of Hudson River samples:

- Laboratory Standard Operating Procedures (SOPs) – Detailed instructions for performing routine laboratory procedures.
- Control charts or data tables – These must be developed and maintained throughout the project for appropriate analyses and measurements.

4.2 LABORATORY SYSTEMS AUDITS

Prior to sample analysis, QA systems audits will be performed. The laboratory audits will be conducted by the QA Coordinator. The checklists used for the laboratory audits are based on requirements outlined in "Good Laboratory Practice Standards" (*40 CFR Part 792*) and audit procedures of the EPA National Enforcement Investigations Center, "NEIC Procedures Manual for the Contract Evidence Audit and Litigation Support for EPA Enforcement Case Development" (*EPA 330/9-89-002*). The Laboratory Project Managers will be informed of the findings and recommendations of the audit before the auditors leave the facility. A written report discussing the audits will be submitted to the Assessment Manager.

Additional laboratory audits may be performed at any time throughout the duration of the NRDA.

4.3 PARTICIPATION IN INTERCOMPARISON EXERCISES

Each analytical laboratory performing PCB analysis is required to participate in the intercomparison exercises for PCBs managed by NIST. A variety of samples including sample extracts and representative matrices (e.g., sediment or tissue samples) are utilized in these exercises, which typically take place once a year. Laboratories are required to analyze the sample(s) in the same manner as specified in this Analytical QA Plan. Laboratories which fail to achieve acceptable performance will be required to provide an explanation to the QA Coordinator and/or undertake appropriate corrective actions.

5.0 ASSESSMENT OF DATA QUALITY

The purpose of this Analytical QA Plan is to develop and document analytical data of known, acceptable, and defensible quality. The quality of the data is presented as a set of statements that describe in precise quantitative terms the level of uncertainty that can be associated with the data without compromising their intended use. These statements are referred to as Data Quality Objectives (DQOs) and are usually expressed in terms of precision, accuracy, completeness, and comparability.

5.1 PRECISION

Precision is the degree of mutual agreement among individual measurements of the same property under prescribed similar conditions, such as replicate measurements of the same sample. Precision is concerned with the "closeness" of the results. Where suitable reference materials (RMs) are available, precision will be expressed as the relative standard deviation (RSD) for the repeated measurements. This use of RMs allows for the long-term measurement of precision but does not include homogenization as a source of analytical variability.

In addition to the tracking precision of replicate RM analyses, precision will be expressed as the relative percent difference (RPD) between a pair of replicate data from duplicate samples.

It is recognized that precision erodes as the limit of detection is approached.

5.2 ACCURACY

Accuracy is the degree of agreement of a measurement with an accepted reference value and may be expressed as the difference between the two measured values or as a percentage of the reference value.

The primary evaluation of accuracy will be through the use of RMs. RMs with certified values (from NIST or a similar source) will be used if they are available. The laboratory will maintain control charts to track the RM performance. Spiked matrix (or ongoing precision and recovery - OPR) samples will also be analyzed to assess accuracy for those analytes that are not available in suitable reference materials.

5.3 COMPARABILITY

Comparability expresses the confidence with which one data set can be evaluated in relationship to another data set. Comparability of the chemical analytical data is established through the use of:

- Program-defined general analytical methodology (e.g., low resolution MS), detection limits, accuracy and precision requirements and reporting formats;
- NIST-traceable calibration materials;
- Reference material with each sample batch.

5.4 COMPLETENESS

Completeness is a measure of the proportion of data specified in the sampling plan which is determined to be valid. Completeness will be assessed by comparing the number of valid sample results to the total number of samples planned for collection. The DQO for completeness is 95%, i.e. no more than 5% of the analytical data missing or qualified as unreliable (rejected).

6.0 QUALITY CONTROL PROCEDURES

No particular analytical methods are specified for this project, but the QA/QC requirements will provide a common foundation for each laboratory's protocols. This "common foundation" includes: (1) the specification of the analytes to be identified and quantified and the minimum sensitivity of the analytical methods and (2) the use of NIST reference materials.

Prior to the analysis of samples, each laboratory must provide written protocols for the analytical methods to be used; calculate detection limits for each analyte in each matrix of interest and establish an initial calibration curve in the appropriate concentration range for each analyte. The laboratory must demonstrate its continued proficiency by participation in refereed intercomparison exercises (PCBs only) and repeated analyses of reference materials, calibration checks, and laboratory method blanks. Laboratories will be expected to take corrective actions promptly if data quality objectives described in this plan are not met.

A laboratory may be audited at any time to determine and document that they have the capability to analyze the samples and can perform the analyses in compliance with the QA plan. Independent data validation will be undertaken promptly after analyses of each sample batch to verify that data quality objectives are met. The data validator will discuss any unacceptable findings with the laboratory as soon as possible, and assist the laboratory in developing a satisfactory solution to the problem.

6.1 Standard Operating Procedures for Analytical Methods

The PCB congeners to be determined are listed in **Table 1.1a**. The target analytes for the additional coplanar PCB congener, dioxin/furan, PBDEs, organochlorine pesticide, and metals analyses are listed in **Tables 1.1b - g**, respectively. Supporting analyses (**Table 1.1h**) include percent solids or moisture (tissue, soil/sediment), percent TEO (tissue), total organic carbon (soil/sediment), and grain size (soil/sediment).

Prior to the analysis of field samples, each laboratory is required to submit to the QA Coordinator for review and approval, written Standard Operating Procedures (SOPs) detailing the procedures used in sample preparation, analysis, data reduction and reporting. Once approved, the SOPs for each analytical method and from each analytical laboratory will be archived with this plan as part of the QA documentation.

6.2 Determination of Method Detection Limit, Quantitation Range, and Reporting Limits

The target detection[4] limits for organics are specified in **Tables 1.1a - f**. It should be noted that the limits provided for metals in **Table 1.1g** are target quantitation[5] limits (not detection limits), and as such are approximately 5 times the expected target detection limit for each element.

The analytical laboratory will establish a method detection limit (MDL) for each analyte of interest in each matrix. The MDLs will be established, for both organics and metals, by following the procedure in 40 CFR 136.

Results greater than the MDL (>MDL) but less than the quantitation limit (<QL) shall be flagged by the laboratory with a J to indicate the result is an estimate. These J-flagged results are not required to meet the DQOs for precision and accuracy because these results will be outside the quantitation range. If the analyte is not detected in the sample, the result will be reported as not detected at the MDL and flagged by the laboratory with a U.

[4] The method detection limit is the lowest concentration that can be detected by an instrument with correction for the effects of sample matrix and method-specific parameters such as sample preparation.

[5] The target quantitation limit is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy (i.e., the DQOs) during routine laboratory operating conditions.

Reporting limits for the supporting analyses (percent moisture, percent TEO, total organic carbon, and grain size) will be 0.01%. The reporting limit will be demonstrated by the laboratory to be greater than 5X the detection limit.

6.3 Quality Control Criteria

The analytical laboratory will determine when control limits (measurement quality objectives) have been exceeded and corrective actions are required before the analyses may proceed. Control limits and required minimum frequency of analysis for each QC element or sample type are summarized in **Tables 6.1a - h**.

6.3.1 Initial Calibration

Acceptable calibration (initial and continuing) must be established and documented before sample analyses may begin. NIST-traceable calibration materials must be used where available in establishing calibration. Initial calibrations will be established according to the criteria in **Tables 6.1a - g**. A specific requirement for this project is to use methodology (and tune instrumentation) for low detection limits, therefore, samples with analytes above the calibration range will be diluted and reanalyzed.

6.3.2 Continuing Calibration Verification

Continuing calibration verification (CCAL) standards will be run at the frequencies indicated in **Tables 6.1a - i**. If CCAL results do not meet the specified criteria, then the instrument must be re-calibrated and all samples analyzed since the last acceptable CCAL must be re-analyzed.

6.3.3 Reference Materials

Reference materials of a matrix appropriate to the samples being analyzed, will be analyzed every 15 samples throughout the analytical program. The data resulting from the analysis of these samples will be reported in the same manner as that from the field samples. These data will be the prime materials used to determine and document the accuracy and precision of the associated field sample data. The following are some of the reference materials that can be used, other reference materials may be used as they become available:

Sediment/Soil (PCB congeners, Dioxin/Furans, Pesticides, Metals)	SRM 1944	New York/New Jersey Waterway Sediment
Tissue (PCB congeners, Pesticides, Metals, moisture, and TEO)	SRM 1974a EDF 2525	Organics in Mussel Tissue Organics in Fish Tissue
Tissue (Dioxin/Furans)	NRCC-CARP-2 EDF 2525	Whole Carp Reference Material Organics in Fish Tissue

TABLE 6.1a

Measurement Quality Objectives for PCB Congeners & Homologues by LRMS
Woods Hole Group

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Calibration	Initially and when continuing calibration (CCAL) fails	Five point curve for all analytes Standard curve percent relative standard deviation (%RSD) < 20% for all analytes except 10% of the analytes may be >20% but <30%
Continuing Calibration [1]	Must start and end analytical sequence and every 12 hours	Percent difference (%D) ≤ 20% for each analyte, up to 10% may be >20% but <30%
GC/MS Tune	Initially and every 72 hours	Within acceptance criteria [2]
Reference Material	Every 15 field samples	Values must be within ±20% of 95% confidence interval for the true value for results greater than 5X the MDL
Method Blank	Every batch (max 15 field samples)	No analytes to exceed 3x MDL unless analyte not detected in associated sample(s) or analyte concentration > 10x blank value
Matrix Spike [3]	Every batch (max 15 field samples)	Percent recovery (%R) = 50% to 125%
Spike Blank [3]	Every batch (max 15 field samples)	%R = 75% to 125%, except up to one analyte may be out
Sample Duplicate [4]	Every batch (max 15 field samples)	RPD ≤ 30% if > 5x MDL
Internal standards	Every sample (added just prior to analysis)	Area of internal standard must be within -50% to +50% of the internal standard from the CCAL at the beginning of the 12 hour sequence
Surrogates	Every sample (added prior to extraction)	%R = 50% to 125%

Notes:

1. %D calculated as follows:

$$\%D = \frac{(\text{True Value} - \text{Calculated Value})}{\text{True Value}} \times 100$$

2. Check instrument tune with a tuning compound (such as DFTPP or PFTBA). Choose three to six ions to check against appropriate acceptance criteria. Criteria should be specified in laboratory standard operating procedures.

3. Spiking solution will contain, at a minimum, one congener from each homologue group.

4. RPD calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2) / 2} \times 100$$

where: C1 is the larger of the duplicate results for a given analyte
C2 is the smaller of the duplicate results for a given analyte

TABLE 6.1b
Measurement Quality Objectives for PCBs (Congeners & Homologues) by LRMS
Axys Analytical

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Calibration	Initially and when continuing calibration (CCAL) fails	Five point curve for all analytes Standard curve percent relative standard deviation (%RSD) < 20% for all analytes except 10% of the analytes may be >20% but <30%
Continuing Calibration [1]	Must start and end analytical sequence and every 12 hours	Percent difference (%D) ≤ 20% for each analyte, up to 10% may be >20% but <30%
GC/MS Tune	Initially and every 12 hours	Within acceptance criteria [2]
Reference Material	Every batch (max 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results greater than 5x the MDL
Method Blank	Every batch (max 15 field samples)	No analytes to exceed 3x MDL unless analyte not detected in associated sample(s) or analyte concentration >10x blank value
Spiked Matrix (OPR) [3]	Every batch (max. 15 field samples)	%R = 50% to 150%, one analyte may exceed these limits
Sample Duplicate [4]	Every batch (max 15 field samples)	RPD ≤ 30% if >5x MDL
Internal standards	Every sample (added just prior to analysis)	Area of internal standard within -50% to +50% of the internal standard from the CCAL at the beginning of the 12 hour sequence
Labeled Compounds	Every sample (added prior to extraction)	%R = 25% to 150%

Notes:

1. %D calculated as follows:

$$\%D = \frac{(\text{True Value} - \text{Calculated Value})}{\text{True Value}} \times 100$$

2. Check instrument tune with a tuning compound (such as DFTPP or PFTBA). Choose three to six ions to check against appropriate acceptance criteria. Criteria should be specified in laboratory standard operating procedures.

3. Spiked Matrix must be performed on a matrix representative of the associated samples. The spiking solution will contain all analytes of interest and will be made from a solution separate from that used for the instrument calibration.

4. RPD calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2) / 2} \times 100$$

where: C1 is the larger of the duplicate results for a given analyte
C2 is the smaller of the duplicate results for a given analyte

TABLE 6.1c

Measurement Quality Objectives for PCB Congeners (including Coplanars and Mono-Ortho Coplanars) by HRMS

Element or Sample Type	Minimum Frequency	Acceptance Criteria
GC/MS Resolution	At the beginning of each 12 hour shift Must start and end each analytical sequence	>10,000 resolving power @ m/z 330.9792 <5 ppm deviation from m/z specified in EPA Method 1668A (EPA 2003)
Initial Calibration	Initially and when continuing calibration fails	Five point curve for toxics/LOC congeners and all labeled compounds. RSD<20% for congeners & <35% for labeled compounds Retention time standard for all congeners Signal to noise ratio (S/N) >10 Ion abundance (IA) ratios within method specified limits [1]
Window Defining / Column Performance Mix [2]	Before every initial and continuing calibration	Valley <40% for PCB34&23 and 187&182 RT of DCBP >55min PCB156/157 must co-elute within 2 sec
Continuing Calibration [3]	At the beginning of each 12 hour shift.	%D <±20% for congeners & <±30% for labeled compounds S/N >10 IA ratios within method specified limits [1] RRT of all compounds within method specified limits [1]
Reference Material	Every batch (max. 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results >5x the method detection limit (MDL)
Method Blank	Every batch (max. 15 field samples)	No analytes to exceed 3x the MDL unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Spiked Matrix (OPR) [4]	Every batch (max. 15 field samples)	%R =50% to 150%, one analyte may exceed these limits
Sample Duplicate (or matrix spike duplicate) [5]	Every batch (max. 15 field samples)	RPD = 30% if value >5x MDL
Labeled Compounds	Every Sample	%R = 15% to 150% (monochlorobiphenyls); 25% to 150% (all others) [1]

Notes:

1. As specified in EPA Method 1668A (EPA 2003).

2. Valley = (X/Y) X 100 where X = height valley and Y = height of shortest peak.

3. Percent difference (%D) = ((True Value - Calculated Value) / True Value) X 100.

4. The spiking solution will contain all analytes of interest and will be made from a solution separate from that used for the instrument calibration, and will be spiked into a representative blank matrix (e.g., tissue, sediment).

5. Relative percent difference (RPD) calculated as follows : RPD= | (C1 - C2) / ((C1 + C2) /2) | X 100. Where C1 is the sample concentration and C2 is the duplicate concentration.

TABLE 6.1d

Measurement Quality Objectives for Dioxin/Furans by HRMS

Element or Sample Type	Minimum Frequency	Acceptance Criteria
GC/MS Tune	At the beginning of each 12 hour shift Must start and end each analytical sequence	>10,000 resolving power @ m/z304.9825 Exact mass of 380.9760 within 5 ppm of theoretical value
Initial Calibration	Initially and when continuing calibration fails	Five point curve for all analytes. RSD<20% for all target compounds & <30% for labeled compounds Signal to noise ratio (S/N) >10 Ion abundance (IA) ratios within method specified limits [1]
Window Defining/Column Performance Mix [2]	Before every initial and continuing calibration	Valley <25% for all peaks near 2378-TCDD/F peaks
Continuing Calibration [3]	Must start and end each analytical sequence	%D <±20% for target compounds & <±30% for labeled compounds S/N >10 IA ratios within method specified limits [1]
Reference Material	Every batch (max. 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results >5x the method detection limit (MDL)
Method Blank	Every batch (max. 15 field samples)	No analytes to exceed 3x the MDL unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Spiked Matrix (OPR) [4]	Every batch (max. 15 field samples)	%R =60% to 160%: one analyte may exceed these limits
Sample Duplicate (or matrix spike duplicate) [5]	Every batch (max. 15 field samples)	RPD ≤ 30% if value >5x MDL
Labeled Compounds	Every sample	%R within method specified limits [6]

Notes:

1. Table 9 of EPA Method 1613B (EPA 1996a).

2. Valley = (X/Y) X 100 where X = height of 2378-TCDD/F and Y = baseline to bottom of valley.

3. Percent difference (%D) = ((True Value - Calculated Value) / True Value) X 100.

4. The spiking solution will contain all analytes of interest and will be made from a solution separate from that used for the instrument calibration, and will be spiked into a representative blank matrix (e.g., tissue, sediment).

5. Relative percent difference (RPD) calculated as follows : RPD= | (C1 - C2) / ((C1 + C2) /2) | X 100. Where C1 is the sample concentration and C2 is the duplicate concentration.

6. Table 7 of EPA Method 1613B (EPA 1996a).

TABLE 6.1e

Measurement Quality Objectives for Organochlorine Pesticides by GC/ECD or GC/MS

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Initial Calibration	On both columns Initially and when continuing calibration fails	Minimum five-point curve for all analytes RSD<20% for target analytes; <30% for surrogates
Breakdown	On both columns With every initial and continuing calibration	DDT and Endrin (Individual) Breakdown: <20%. Combined Breakdown: <30%. Compounds within retention time windows (RTW) [1]
Continuing Calibration [2]	On both columns At the beginning and end of each 12 hour shift	%D <±25% for analytes and surrogates. Compounds within retention time windows (RTW) [1]
GC/MS Tune	Initially and every 12 hours	Within acceptance criteria [3]
Reference Material	Every batch (max. 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results >5x the method detection limit (MDL)
Method Blank	Every batch (max. 15 field samples)	No analytes to exceed 3x the MDL unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Spiked Matrix (OPR) [4]	Every batch (max. 15 field samples)	%R = 70% to 130%
Sample Duplicate (or matrix spike duplicate) [5]	Every batch (max. 15 field samples)	RPD ≤ 30% if value >5x MDL
Labeled Compounds (or Surrogates)	Every sample	%R = 30% to 130%

Notes:

1. As specified in EPA Method 8000B (EPA 1996b), or equivalent.

2. Percent difference (%D) = ((True Value - Calculated Value) / True Value) X 100.

3. Criteria should be specified in laboratory standard operating procedures. Does not apply to GC/ECD analyses.

4. The spiking solution will contain all analytes of interest and will be made from a solution separate from that used for the instrument calibration, and will be spiked into representative blank matrix (e.g., tissue, sediment).

5. Relative percent difference (RPD) calculated as follows : RPD= | (C1 - C2) / ((C1 + C2) /2) | X 100. Where C1 is the sample concentration and C2 is the duplicate concentration.

TABLE 6.1f

Measurement Quality Objectives for PBDEs by HRMS

Element or Sample Type	Minimum Frequency	Acceptance Criteria
GC/MS Resolution	At the beginning of each 12 hour shift Must start and end each analytical sequence	>10,000 resolving power @ m/z 330.9792 <5 ppm deviation from m/z specified in EPA Method 1668A (EPA 2003)
Initial Calibration	Initially and when continuing calibration fails	Five point curve for analytes and all labeled compounds RSD<20% for analytes & <35% for labeled compounds (<100% for labeled DecaBDE) Retention time standard for analytes Signal to noise ratio (S/N) >10 Ion abundance (IA) ratios within method specified limits
Continuing Calibration [1]	At the beginning of each 12 hour shift	%D <±25% for analytes & <±35% for labeled compounds S/N >10 IA ratios within method specified limits RRT of all compounds within method specified limits
Reference Material	Every batch (max. 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results >5x the method detection limit (MDL)
Method Blank	Every batch (max. 15 field samples)	No analytes to exceed 3x the target DL unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Spiked Matrix (OPR) [2]	Every batch (max. 15 field samples)	%R = 50% to 150%, one analyte may exceed these limits
Sample Duplicate [3]	Every batch (max. 15 field samples)	RPD ≤ 30% if value >5x MDL
Labeled Compounds	Every sample	%R = 25% to 150% (Labeled DecaBDE = 20-200%)

Notes:

1. Percent difference (%D) = ((True Value - Calculated Value) / True Value) X 100.

2. Spiked Matrix must be performed on a matrix representative of the associated samples. The spiking solution will contain all analytes of interest and will be made from a solution separate from that used for the instrument calibration.

3. Relative percent difference (RPD) calculated as follows : RPD= | (C1 - C2) / ((C1 + C2) /2) | X 100. Where C1 is the sample concentration and C2 is the duplicate concentration.

TABLE 6.1g

Measurement Quality Objectives for Metals by ICP/ICP-MS and Mercury by CVAA

Element or Sample Type	Minimum Frequency	Acceptance Criteria
ICP-MS Tune	At the beginning of each 24 hour shift Must start each analytical sequence	Analyze 4 times with RSD<5% Resolution 0.9 amu at 10% peak height
Initial Calibration	Initially and when continuing calibration fails	Minimum of a 2 point curve for ICP/ICP-MS 5 point curve for Mercury by cold vapor atomic absorption (CVAA) Linear regression correlation coefficient r>0.995 for curves with more than two points
Independent Calibration Verification (ICV)	Analyzed immediately after calibration and prior to samples	Different source than calibration standards Percent recovery (%R)[1] 90% to 110% ICP/ ICP-MS %R = 80% to 120% Mercury
Continuing Calibration Verification	Must be analyzed before samples, after every 10 samples, and end each analytical sequence	%R = 90% to 110% ICP/ICP-MS %R = 80% to 120% Mercury
Continuing Calibration Blanks (CCB)	Must be analyzed after each continuing calibration verification (CCV)	No analytes to exceed the quantitation limit unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Reference Material	Every batch (max. 15 field samples)	Values must be within ±20% of 95% confidence interval for the true value for results >5x the method detection limit (MDL)
Method Blank	Every batch (max. 15 field samples)	No analytes to exceed the quantitation limit unless analyte not detected in associated sample(s) or analyte concentration >10x the blank value
Matrix Spike	Every batch (max. 15 field samples)	%R[2] = 75% to 125%
Sample Duplicate (or matrix spike duplicate) [3]	Every batch (max. 15 field samples)	RPD ≤ 20% if value >5x MDL
Internal Standards (ICP-MS only)	Every sample	%R = 50% to 120%

Notes:

1. Percent recovery (%R) = ((Found Value) / True Value) X 100.

2. %R = ((MS conc - sample conc)/spike amount) X 100.

3. Relative percent difference (RPD) calculated as follows : RPD= | (C1 - C2) / ((C1 + C2) /2) | X 100. Where C1 is the sample concentration and C2 is the duplicate concentration.

TABLE 6.1h

Percent Solids, Moisture, Percent TEO, and Grain Size[1]

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Duplicates [2]	Every 15 field samples	RPD ≤ 15%
Reference Material	Every 15 field samples	Value must be within ±20% of 95% confidence interval for the true value

Notes:

1. Grain size: Five fractions (gravel, coarse sand, medium sand, very fine sand, and silt/clay).

2. RPD calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2) / 2} \times 100$$

where: C1 is the larger of the duplicate results for a given analyte
C2 is the smaller of the duplicate results for a given analyte

TABLE 6.1i

Total Organic Carbon (TOC)

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Continuing Calibration [1]	Must start and end analytical sequence and every 10 samples	%D ≤ 10%
Method Blank	Every batch (max 15 field samples)	Not to exceed MDL
Reference Material	Every batch (max 15 field samples)	Values must be within ±20% of 95% confidence interval
Triplicate	Every 15 field samples	RSD ≤ 15%

Notes:

1. %D calculated as follows:

$$\%D = \frac{(\text{True Value} - \text{Calculated Value})}{\text{True Value}} \times 100$$

It is recognized that absolute accuracy can only be assessed using certified values, hence the term relative accuracy. Relative accuracy is computed by comparing the laboratory's value for each analyte against either end of the range of values (i.e., 95% confidence limits) reported by the certifying agency. The laboratory's value must be within 20% of either the upper or lower 95% confidence interval value. For commercial reference materials (e.g. EDF 2525), the acceptance range may be derived from laboratory control limits if the QA Coordinator deems the certified range is too wide to provide reasonable accuracy. Non-certified results can be compared, but with less rigorous criteria.

Accuracy control limit criteria (**Tables 6.1a - i**) will apply for analytes having concentrations greater than 5 times the laboratory's MDL. Each laboratory will record the results for each analyte on control charts. In the case of analytes for which no concentration information is provided, the laboratory will establish upper and lower control limits, based on three standard deviations of the mean. These control limits will be evaluated on a monthly basis.

6.3.4 Method (Reagent) Blanks

Method blanks are laboratory derived samples which have been subjected to the same preparation or extraction procedures and analytical protocols as project samples. A method blank will be analyzed with every 15 field samples analyzed. Acceptance criteria are provided in **Tables 6.1a - i**. Failure to meet acceptance criteria requires definitive corrective action to identify and eliminate the source(s) of contamination before the subsequent reanalysis and re-extraction of the blank and affected samples. Sample results will not be blank corrected.

6.3.5 Sample Duplicates

A duplicate sample aliquot from a representative matrix will be prepared and analyzed with every 15 field samples. Acceptance criteria are provided in **Tables 6.1a - i**.

6.3.6 Matrix Spike

Matrix spikes (MS) will be analyzed every 15 samples unless an isotope dilution method is used (i.e., each sample is spiked with labeled compounds). Samples will be spiked prior to extraction. Spike solution concentrations for the MS must be appropriate to the matrix and anticipated range of contaminants in the sample; that is 2 to 10 times analyte concentration. However, because it is not possible to know the concentration of contaminants prior to analysis, professional judgment may be exercised in choosing concentrations that are reasonable under the circumstances.

6.3.7 Spiked Matrix Blanks

Spiked matrix blanks will be analyzed every 15 samples. Extraction solvent will be spiked and handled in the same manner as the sample. Spike solution concentrations for the spike blank must be appropriate to the matrix and anticipated range of contaminants in the sample. However, because it is not possible to know the concentration of contaminants prior to analysis, professional judgment may be exercised in choosing concentrations that are reasonable under the circumstances.

6.3.8 Internal Standards

All samples will be spiked with internal standards prior to analysis, when required by the analytical method. Control criteria for internal standard recovery are listed in **Tables 6.1a and b**.

7.0 DATA REDUCTION, VALIDATION AND REPORTING

7.1 DATA REDUCTION

Data reduction is the process whereby raw data (analytical measurements) are converted or reduced into meaningful results (analyte concentrations). This process may be either manual or electronic. Primary data reduction requires accounting for specific sample preparations, sample volume (or weight) analyzed, and any concentrations or dilutions required.

Primary data reduction is the responsibility of the analyst conducting the analytical measurement and is subject to further review by laboratory staff, the Laboratory Project Manager and finally, independent reviewers. All data reduction procedures will be described in the laboratory SOPs.

- Concentrations will be reported as if three figures were significant.
- Organic analytes in sediments will be reported in ng/g, dry weight.
- Organic analytes in tissues will be reported in ng/g, wet weight.
- Data generated from the analysis of blank samples will not be utilized for correction of analyte data.
- Surrogate compounds, matrix spikes, and spike blanks will be evaluated as %R.
- Reference materials will be reported in units indicated on the certificate of analysis.
- Continuing calibration factors will be presented as %D for organic analyses and %R for inorganic analyses.
- Duplicate sample results will be expressed as RPD.
- PCB homologue totals will be calculated as follows: first, the concentrations of all target congeners that meet the identification acceptance criteria will be calculated. Next, each remaining peak will be evaluated to determine if it meets the identification acceptance criteria for a PCB congener (criteria will be specified in the laboratory SOP). If the criteria are met, these peaks will be included as the other non-target congeners within the appropriate homologue group. [The ICAL will contain at least one peak in each homologue group, and the concentrations of the non-target congeners will be determined using a representative response factor from the ICAL.] If a peak does not meet the identification criteria, the peak is not included in the summation. The total for each homologue group will be obtained by summing all target and non-target congener concentrations within each homologue group. If a congener is reported as non-detected, then zero will be used in the summation.
- Total PCBs are calculated by summing the concentrations of PCB homologues. If a result is reported as non-detected, then zero will be used in the summation (which will minimize the potential for high bias).

7.2 DATA REVIEW AND VALIDATION

Data review is an internal review process where data are reviewed and evaluated by personnel within the laboratory. Data validation is an independent review process conducted by personnel not associated with data collection and generation activities.

Data review is initiated at the bench level by the analyst, who is responsible for ensuring that the analytical data are correct and complete, the appropriate SOPs have been followed, and the QC results are within the acceptable limits. The Laboratory Project Manager has final review authority. It is the Laboratory Project Manager's responsibility to ensure that all analyses performed by that laboratory are correct, complete, and meet project data quality objectives.

External and independent data validation will be performed for all samples by the QA Contractor using a data package (**Table 7.1**) containing sufficient information to allow the independent validation of the sample identity and integrity, the laboratory measurement system, and resulting quantitative and qualitative data.

Three levels of data validation will be performed: full, summary, or cursory validation. Full validation will consist of a review of the entire data package for compliance with documentation and quality control criteria for all the following items, plus recalculations of instrument calibration curves, sample and QC results. Summary validation will consist of a review of all the following items, but without recalculations. Cursory validation will consist of a review of only the starred (*) items:

- Package completeness*
- Holding times from extraction to analysis*
- Instrument calibration, initial and continuing
- Blank results*
- Instrument performance
- Spike recoveries (PCBs only)*
- Standard reference material results*
- Laboratory duplicate results*
- Reported detection limits*
- Compound quantitation
- Verification of electronic data deliverable (EDD) against hardcopy (10% verification)*

TABLE 7.1
Laboratory Data Deliverables Per Sample Batch

Chain-of-Custody/Sample Receipt Checklist	
Sample Data:	Result summaries including surrogate recoveries, percent total solids, dilutions, etc.
Standards Data:	Target MDL data based on the method in 40 CFR, 136, or data from analyses of SRM which has analytes at low concentrations (submitted once each year for each laboratory/matrix). Calibration summaries: Initial calibration data, standard curve equation, correlation coefficient or %RSD, continuing calibration %D.
Quality Control Data (Method Blanks, CRMs, Duplicates, Matrix Spikes, Spike Blanks):	Results summaries including surrogate recoveries, plus %R and RPD, as applicable.
Case Narrative:	Special handling or analysis conditions. Any circumstance that requires special explanation such as an exception to QA/QC conditions or control criteria, dilutions, reanalysis, etc. Corrective actions/procedure alterations.
Electronic Data Deliverable:	As specified in laboratory contract.

As the project proceeds and the quality of the data is verified and documented, the level of validation will decrease at the discretion of the QA Coordinator. At a minimum, cursory validation will be performed on all data packages, i.e., only the starred items will be reviewed.

Qualifiers (**Table 7.2**) may be assigned to individual data points by the QA Contractor. These validation qualifiers will not replace qualifiers or footnotes provided by the laboratory, but will be added to the data summary tables to inform the data user whether or not the data met all project quality objectives. Both sets of qualifiers will be maintained in the database.

TABLE 7.2
Data Validation Qualifier Codes

U	Analyte concentration is not significantly greater than the associated blank result. The result is judged to be the detection limit.
R	Unreliable result. Data should not be used.
J	Reported concentration may not be accurate or precise, as judged by associated calibration and/or reference material results.
UJ	Not detected. Detection limit may be inaccurate or imprecise, as judged by the associated quality control results.

All discrepancies and requests for additional corrected data will be discussed with the laboratory prior to issuing the formal data validation report. Review procedures and findings during data validation will be documented on worksheets. A validation report will be prepared for each data group/data package summarizing QC results, qualifiers, and possible data limitations. Only validated data with appropriate qualifiers will be released for general use. Data are not considered final until the QA Coordinator has performed assessment and accepted the data.

8.0 CORRECTIVE ACTION/PROCEDURE ALTERATION

The analytical laboratories are required to adhere to the SOPs submitted by them to the QA Coordinator for this project. When the data from the analyses of any quality control sample exceeds the project specified control limits or indicates that the analytical method is drifting out of control, it is the immediate responsibility of the analyst to identify and correct the situation before continuing with sample analysis.

A narrative describing the problem noted, the steps taken to identify and correct the problem and the treatment of the relevant sample batches must be prepared and submitted with the relevant data package. If the action is a change from the accepted SOP, the SOP must be revised and re-submitted within 30 working days after the problem was noted.

9.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality Assurance/Quality Control (QA/QC) reports will be submitted periodically to the Assessment Managers by the QA Coordinator. These reports may be either formal or informal in response to the Assessment Manager's request. Upon termination of the analytical work for this damage assessment, a formal QA report will be submitted. This report will include:

- General compliance with QA objectives
- Summary of technical and performance evaluation audits
- Summary of data validation reports
- Summary of laboratory control charts

10.0 REFERENCES

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