Quality Assurance Plan for Analyses of Environmental Samples

for Polycyclic Aromatic Compounds,
Persistent Organic Pollutants, Fatty Acids,
Stable Isotope Ratios, Lipid Classes, and
Metabolites of Polycyclic Aromatic Compounds

July 2006

U.S. DEPARTMENT OF COMMERCE
National Oceanic and Atmospheric Administration
National Marine Fisheries Service

NOAA Technical Memorandum
NMFS Series

NOAA Technical Memorandum NMFS-NWFSC-77

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Executive Summary

This technical memorandum serves as the Quality Assurance Plan (QAP) for analyses of marine biota and sediment samples by the Environmental Assessment Program (EAP) within the Environmental Conservation Division at the Northwest Fisheries Science Center. This QAP describes the EAP's quality objectives, as well as policies implemented for achieving the objectives and procedures for assessing the completeness of the objectives. It also provides guidelines for monitoring and documenting the quality of analyses so that a desired level of performance can be demonstrated and maintained. These guidelines are based on protocols established previously for specific projects under the National Oceanic and Atmospheric Administration and the Environmental Protection Agency and have been adapted to new types of analyses and current technologies.

Abbreviations and Acronyms

CM	control material
COC	chain of custody
CCV	continuing calibration verification
DDT	dichlorodiphenyltrichloroethane
DQO	data quality objectives
EA/IRMS	elemental analyzer/isotope ratio mass spectrometry
EAP	Environmental Assessment Program
GC/MS	gas chromatography/mass spectrometry
GLP	good laboratory practices
HPLC/PDA	high-performance liquid chromatography/photodiode array
IAEA	International Atomic Energy Agency
IRM	interim reference material
LOQ	limit of quantitation
mV	millivolt
NIST	National Institute of Standards and Technology
PAC	polycyclic aromatic compound
PBDE	polybrominated diphenyl ether
PCB	polychlorinated biphenyl
POP	persistent organic pollutant
QA	quality assurance
QAP	quality assurance plan
RSD	relative standard deviation
SRM	Standard Reference Material
SOP	standard operating procedure
TLC/FID	thin-layer chromatography/flame ionization detection

1.0. Introduction

The Environmental Assessment Program (EAP) within the Environmental Conservation Division at the Northwest Fisheries Science Center has developed a Quality Assurance Plan (QAP) for analyses of marine biota and sediment samples. Many of the requirements described in the QAP, this technical memorandum, are based on protocols that were originally developed for programs under the National Oceanic and Atmospheric Administration (National Status and Trends Program and Natural Resource Damage Assessment) and the Environmental Protection Agency (Puget Sound Estuary Program and Environmental Monitoring and Assessment Program-Estuaries). These programs were designed to measure low-level (i.e., low parts per billion) concentrations of contaminants in marine and estuarine sediments and biota using gas chromatography/mass spectrometry (GC/MS) and high-performance liquid chromatography/photodiode array (HPLC/PDA). More recently the EAP has expanded its studies to include analyses of samples for fatty acids by GC/MS and for stable isotope ratios by elemental analyzer/isotope ratio mass spectrometry (EA/IRMS). Other analyses performed by the EAP include determining lipid classes in tissues using thin-layer chromatography/flame ionization detection (TLC/FID) and measuring relative amounts of metabolites of polycyclic aromatic compounds (PACs) in bile by HPLC/fluorescence.

The requirements specified in this QAP are designed to:

1) monitor the performance of the measurement systems to maintain quality, and

2) document the extent to which the reported data are sufficiently complete, comparable, representative, unbiased, and precise to be suitable for their intended use.

This QAP will be adapted to specific projects as needed, and will be revised as appropriate as changes are made to the EAP quality assurance program.

The term "field samples" in this technical memorandum refers to samples collected from the environment (e.g., sediments, plant or animal tissues). The term "samples" refers to field samples or quality assurance (QA) samples. QA samples are analyzed concurrently with the field samples using the same method. The established methods used to measure the various groups of analytes (i.e., contaminants, fatty acids, stable isotope ratios, lipids, or metabolites) are documented separately and are available in the form of standard operating procedures (SOPs). In addition this QAP does not address the collection of samples; that will be addressed in a separate document (e.g., a sampling plan), when appropriate.

2.0. Project Description

The description of a specific project is to be provided before the analyses begin in order to ensure that the project requirements are known and can be met. The project description may include the following information:

- the principal investigator(s),
- the project's objectives, questions, or issues,
- what type, quantity, and quality of analyses are required,
- timeframe for receipt of samples, analyses, and data delivery,
- how the results are to be formatted and reported,
- who will use the data, and
- what decision(s) will be made from the information obtained.

A project proposal that includes the information in a project description may be used for this purpose.

3.0. Laboratory Organization and Responsibilities

The analyses for the project will be performed primarily by personnel from the Environmental Conservation Division of the Northwest Fisheries Science Center.

3.1. Project Leader

Dr. Margaret (Peggy) Krahn, EAP manager, is responsible for ensuring that the analytical data quality objectives (DQOs) for the project are met and that staff resources are available to fulfill laboratory analytical requirements. Her contact information is:

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3.2. Analytical Laboratory Project Managers

Donald Brown, Catherine Sloan, and Gina Ylitalo, EAP team leaders, are responsible for ensuring that the analytical results meet QA criteria and the stated objectives. Their mailing address and fax number is the same as for the project leader. Their individual contact information is:

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4.0. Sample and Data Handling

Sample handling procedures may be dependent on the matrix and the analytes of concern. The groups of analytes that can be determined by the EAP laboratory are presented in Tables 1–7; the analytes within each group are measured concurrently using the specified method. Sampling procedures, including sample collection, preservation, storage, and documentation, are addressed in detail elsewhere (e.g., in a sampling and analysis plan). In general, samples and data are handled according to the following steps:

1) inventory all samples received,
2) store the samples in freezers prior to analyses,
3) record the sample identification, information, and storage location in a sample database,
4) schedule the batches of samples to be analyzed and prepare tracking paperwork,
5) analyze the batches of samples for the specified analytes,
6) process the raw sample data,
7) review the processed and formatted data,
8) report the reviewed data,
9) archive all remaining sample material in freezers, and
10) archive the raw and processed sample data.

Chain-of-custody (COC) procedures (subsection 4.5, Chain of Custody, page 8), if required by the project, will be followed for all field samples throughout the sampling and analytical process.

4.1. Intralaboratory Sample Transfer

The laboratory analysts will maintain a laboratory sample-tracking record, similar to the COC record that will follow each sample through all stages of laboratory processing. The sample-tracking record will show the date of sample extraction or preparation and sample analysis, as well as the names or initials of individuals responsible for each procedure.

4.2. Sample Archival

All unanalyzed samples and unused sample aliquots or extracts will be held by the laboratory in a manner to preserve sample integrity (e.g., at –20°C to –80°C) for at least one year or a specified time period after the data have been validated, as agreed upon by the project leader and the principal investigator(s).

Table 1. Polycyclic aromatic compounds (PACs) determined by gas chromatography/mass spectrometry
(GC/MS).* Method: Sloan et al. 2004 and 2005.

Low molecular weight PACs	High molecular weight PACs
Naphthalene	Fluoranthene
1-Methylnaphthalene	Pyrene
2-Methylnaphthalene	Benz[*a*]anthracene
Biphenyl	Chrysene + triphenylene
2,6-Dimethylnaphthalene	Benzo[*b*]fluoranthene
Acenaphthylene	Benzo[*j*]fluoranthene + Benzo[*k*]fluoranthene
Acenaphthene	Benzo[*e*]pyrene
2,3,5-Trimethylnaphthalene	Benzo[*a*]pyrene
Fluorene	Perylene
Dibenzothiophene	Indeno[1,2,3-*c*,*d*]pyrene
Phenanthrene	Dibenz[*a*,*h*]anthracene + Dibenz[*a*,*c*]anthracene
Anthracene	Benzo[*ghi*]perylene
1-Methylphenanthrene	HPACs (sum of high molecular weight PACs)
Retene	
LPAC (sum of low molecular weight PACs)	

* Sums of alkylated PACs (from petroleum sources) also can be determined, when requested.

4.3. Laboratory Records

Laboratory log books will be maintained for each of the following:

- sample preparation,
- use and maintenance of the accelerated solvent extractors,
- use and maintenance of the HPLC,
- use and maintenance of the GC/MS,
- use and maintenance of the EA/IRMS, and
- use and maintenance of the TLC/FID.

Final analytical results will be generated and maintained in electronic database files with frequent backup and storage on permanent media.

4.4. Data and Data Documentation

The laboratory will provide data tables and QA documentation suitable for QA assessment. All original data and data documentation developed by the laboratory for a given data package will be kept by the laboratory for at least one year after the data have been validated and reported; and if requested, the data will be stored in the collection format for up to five years.

Table 2. Persistent organic pollutants (POPs) determined by GC/MS. Method: Sloan et al. 2004 and
2005.

Polychlorinated biphenyl (PCB) congeners[a] (by IUPAC number) and estimated total PCBs[b]	Dichlorodiphenyltrichloroethanes (DDTs)
17, 18,* 28,* 31, 33, 44,* 49, 52,* 66,* 70, 74, 82, 87, 95, 99, 101/90,* 105,* 110, 118,* 128,* 138/163/164,* 149, 151, 153/132,* 156, 158, 170/190,* 171, 177, 180,* 183, 187,* 191, 194, 195,* 199, 205, 206,* 208, 209,*	2,4'-DDT, 4,4'-DDT, 2,4'-DDD, 4,4'-DDD, 2,4'-DDE, 4,4'-DDE
Polybrominated diphenyl ether (PBDE) congeners (by IUPAC number)	**Other organochlorine pesticides:**
28, 47, 49, 66, 85, 99, 100, 153, 154, 183	Aldrin
	cis-Chlordane
	trans-Chlordane
	Dieldrin
	Endosulfan I
	Hepatchlor
	Heptachlor epoxide
	Hexachlorobenzene
	alpha-Hexachlorocyclohexane
	beta-Hexachlorocyclohexane
	gamma-Hexachlorocyclohexane
	Mirex
	cis-Nonachlor
	trans-Nonachlor
	Nonachlor III
	Oxychlordane

[a] For the PCBs, 46 congeners present in 40 chromatographic peaks are measured (listed with coeluting congeners).

[b] Estimated total PCB concentrations: calculated as 2 × the sum of concentrations of 17 congeners noted * (NOAA 1989) or as the sum of concentrations of all 40 congeners measured.

Table 3. POPs determined by high-performance liquid chromatography/photodiode array detection
(HPLC/PDA). Methods: Krahn et al. 1994 and Ylitalo et al. 2005a for tissues and Buzitis et al. in
press for sediments.

PCB[a] congeners (by IUPAC number) and estimated total PCBs[b]:	DDTs and other pesticides
77, 101,* 105,* 110,* 118, 126, 128,* 138, 153,* 156, 157,* 169, 170/194,* 180, 189, 190, 200	2,4'-DDT, 4,4'-DDT, 2,4'-DDD, 4,4'-DDD, 4,4'-DDE

[a] The peaks represented by the analytes noted * may contain coeluting compounds.

[b] Estimated total PCBs: calculated by summing the concentrations of identified PCB congeners above then adding the concentration of the remaining congeners, which have been calculated by summing their PDA response areas and applying an average response factor.

Table 4. Fatty acids determined as methyl ethers by GC/MS.[a] Method: Krahn et al. 2004.

Fatty acids (quantitative)[b]	Tentatively identified fatty acids (semiquantitative)[c]
C10:0 (capric acid)	C14:1n9
C11:0	4,8,12-trimethyl-C13:0
C12:0 (lauric acid)	C14:1n7
C12:1	11-methyl-C14:0
iso-C14:0	anteiso-C15:0
C14:0 (myristic acid)	anteiso-C16:0
C14:1n5	2,6,10,14-tetramethyl-C15:0
iso-C15:0	C16:1n11 (may coelute with C16:1n12)
C15:0	C16:1n9
C15:1n5	C16:1n5
iso-C16:0	7-methyl-C16:1
C16:0 (palmitic acid)	C16:2n6
C16:1n7 (palmitoleic acid)	anteiso-C17:0
iso-C17:0	7,8-dimethyl-C16:1
C17:0 (margaric acid)	C16:2n4
C17:1n7	C16:3n6
iso-C18:0	C16:3n4
C18:0 (stearic acid)	C17:1n8 (tentatively identified structural isomer)
C18:1n9 (oleic acid)	C16:4n3
C18:1n7 (vaccenic acid)	anteiso-C18:0
C18:2n6 (linoleic acid)	C16:4n11
C19:0	C18:1n13
C18:3n6 (gamma-linolenic acid)	C18:1n11 (may coelute with C18:1n12)
C18:3n3 (alpha-linolenic acid0	C18:1n5
C18:4n3 (stearidonic acid)	C18:2n7
C20:0 (arachidic acid)	C18:2n4
C20:1n15	C18:3n4
C20:1n9 (gadoleic acid)	C18:3n1
C20:2n6	C18:4n
C20:3n6	C20:1n7
C20:4n6 (arachidonic acid)	C20:1n5
C20:3n3	C20:1n11 (may coelute with C20:1n12)
C20:5n3	C20:2n11
C22:0 (behenic acid)	C20:2n9
C22:1n9 (erucic acid)	C20:4n3
C22:2n6	C22:1n11 (may coelute with C22:1n12)
C22:4n6	C22:1n7
C22:3n3	C22:1n5
C24:0	C21:5n3
C22:5n3	C22:4n3
C22:6n3	
C24:1n9 (nervonic acid)	

[a] All analytes are the isomers in the *cis* configuration.

[b] Quantitation uses the relative response factor of the analyte.

[c] Quantitation uses the relative response factor of the most similar homolog for which there is a standard.

Table 5. Stable isotope ratios determined by elemental analyzer/isotope ratio mass spectrometry
(EA/IRMS). Method: Krahn et al. in prep.

Delta (δ) values	Other ratios
δ13C, δ15N	Percent nitrogen by weight (Wt %N), percent carbon by weight (Wt %C), carbon/nitrogen ratio (C/N ratio)

Table 6. Lipid class proportions and percent lipid determined by thin-layer chromatography/flame
ionization detection (TLC/FID). Method: Krahn et al. 2001, Ylitalo et al. 2005b.

Lipid classes	Percent lipid
Sterol/wax esters Triglycerides Free fatty acids Cholesterol/sterols Phospholipids/other polar lipids	Percent lipid determined by summing the concentrations (g of lipid class/kg sample) of the five lipid classes and multiplying this sum by 0.1%

Table 7. Metabolites of PACs determined by high-performance liquid chromatography
(HPLC)/fluorescence. Method: Krahn et al. 1987.

PAC equivalents
Phenanthrene equivalents
Benzo(*a*)pyrene equivalents
Naphthalene equivalents

4.5. Chain of Custody

When COC records are required, each field sample will be assigned a unique identification number and will have a separate entry on the COC record. COC records will be completed with indelible ink. A sample is considered "in custody" if:

- it is in the custodian's actual possession or view,
- 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 such that the sample cannot be reached without breaking the seal.

Samples are kept in the custody of the designated sampling or field personnel or both until shipment. Any transfer or movement of samples will use COC procedures. Samples will be properly packaged for shipment near the sampling area and dispatched to the appropriate party. The original signed and dated COC record will accompany the sample(s); a copy of the COC record is retained by the sample shipper. All shipments will comply with Department of Transportation regulations (49 CFR, parts 172 and 173). Immediately upon receipt of samples,

the recipient will review the shipment for sample condition and consistency with the accompanying COC record before signing and dating the COC record. Sample condition(s) will be noted on the original COC sheet at this time. If there are any discrepancies between the COC record and the sample shipment, the recipient will contact the sample shipper immediately.

5.0. Assessment of Data Quality

The overall QA objectives are to ensure development of analytical data of known and acceptable quality. The quality of data required is specified in qualitative and quantitative DQOs. These objectives usually are expressed in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity. Data quality is assessed by applying the specific acceptance criteria to QA elements (section 6.0, Quality Assurance Procedures, page 12).

5.1. Precision

Precision is the degree of agreement among individual measurements of the same property under prescribed similar conditions (e.g., replicate measurements of a particular analyte in one sample). Laboratory precision is evaluated using laboratory replicates of field samples and Standard Reference Materials (SRMs) when available (subsection 6.5.6, Sample Replicates, page 23). The use of SRMs allows for the long-term measurement of precision, whereas replicates of field samples can indicate the precision for a particular group of samples. Precision will be expressed as the relative standard deviation (RSD) for repeated measurements. The RSD is an estimate of the average standard error in a measurement; this estimate generally improves with increasing number of replicates. Reproducibility is affected by sample collection procedures and matrix variations, as well as the extraction and analytical procedures used. It is recognized that, typically, precision erodes as the limit of detection is approached.

5.2. Accuracy

Accuracy is the degree of agreement of a measurement with an accepted (e.g., certified or published) value. Laboratory accuracy will be evaluated through the use of SRMs when available (subsection 6.5.3, Reference Materials, page 21). For a particular SRM, accuracy for an analyte will be assessed by comparing the measured value to a value accepted (i.e., certified or published) by the certifying agency (i.e., the National Institute of Standards and Technology [NIST]) (subsection 6.5.3, Reference Materials, page 21).

5.3. Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a defined or particular characteristic of a population, parameter variations at a sampling point, a processed condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program (as addressed in a sampling plan) and proper laboratory protocol. Evaluation of the data for reference materials and replicate field samples may provide an assessment of the representativeness of the analyte measurements for field samples (subsections 6.5.3, Reference Materials, page 21, and 6.5.6, Sample Replicates, page 23).

5.4. Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another, as well as the potential for combining the data with those generated outside of the present project. Comparability of the analytical data is established through the use of the following:

- program-defined analytical methodology, quantitation limits, reporting units, and quality assurance measurements,
- NIST-traceable (or other) calibration standards and SRMs, when available, and
- participation in interlaboratory comparison exercises (subsection 6.3, Participation in Intercomparison Exercises, page 13).

5.5. Completeness

Completeness is defined as the percentage of measured data that meet the DQOs as determined by the QA review process. A typical analytical completeness goal for a project is 90% (i.e., no more than 10% of the analytical data will be qualified as unreliable; meaning it does not meet the DQOs). Data qualified as estimated as a result of QA criteria not being met will be considered usable.

5.6. Sensitivity

Sensitivity refers to the capability of a method to measure the analytes at low levels. For each method, criteria are established for the minimum concentrations that can be measured with known and acceptable quality (subsection 6.4, Quantitation Range, page 13).

6.0. Quality Assurance Procedures

Prior to the analysis of samples, the laboratory will specify written protocols for the analytical methods to be used and will identify the analytes to be quantified. If a method is significantly modified, the written analytical protocol will be amended. QA procedures are presented with each analytical method and are applied to each batch (i.e., group of field samples and QA samples analyzed concurrently). The laboratory also must demonstrate its continued proficiency by participation in refereed intercomparison exercises, as available. The QA procedures and criteria presented in this document may be specific to the protocols and instrumentation currently in use by the EAP.

6.1. Laboratory Operations

The laboratory will have the appropriate facilities to store and prepare samples and the appropriate instrumentation and staff to provide data of the required quality within the time period indicated. The laboratory is expected to conduct operations using good laboratory practices (GLP), including:

- performing scheduled maintenance of analytical balances, laboratory equipment, and instrumentation,

- validating instrument calibration standards, and

- recording pertinent analytical data in logbooks with each entry signed and dated by the analyst.

Personnel should be well versed in GLP, including standard safety procedures. It is the responsibility of the project manager to ensure that mandatory safety training is completed by all laboratory personnel. The laboratory is responsible for maintaining a current safety manual in compliance with the Occupational Safety and Health Administration 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 appropriately based on its potential health hazard.

6.2. Quality Assurance Documentation

All participants in a project must have the current version of the QAP. In addition the following documents and information must be current and available to all laboratory personnel participating in the processing of samples:

- laboratory SOPs—the detailed instructions for performing routine laboratory procedures,

- instrument performance information—for example, information on instrument calibration, range of response and stability, and

- QA information—QA tables will be developed and maintained throughout the project for all appropriate analyses and measurements.

The SOPs used in the analyses of samples depend on the project and the analytes to be determined (listed in Tables 1–7). Documentation of all analytical methods will accompany the analytical results.

6.3. Participation in Intercomparison Exercises

The analytical laboratory is required to participate, whenever possible, in the intercomparison exercises managed by the NIST or the International Atomic Energy Agency (IAEA). A variety of samples including accuracy-based solutions, sample extracts, and representative matrices (e.g., sediment or tissue samples) is used in these exercises, which typically take place once a year. Upon review of the results, if the laboratory fails to achieve acceptable performance, it will be required to undertake appropriate corrective actions. This section applies only to analyses for PACs (Table 1) and POPs (Tables 2 and 3); it does not apply to analyses for fatty acids (Table 4), carbon and nitrogen stable isotope ratios (Table 5), lipid classes (Table 6), or metabolites of PACs (Table 7) because formal intercomparison exercises by the NIST or the IAEA have not been conducted for these analytes. Acceptance criteria are the same as those for reference materials (subsection 6.5.3, Reference Materials, page 21).

6.4. Quantitation Range

For each GC/MS method (for PACs, POPs, and fatty acids), the lower limit of quantitation (LOQ) for a given analyte in a specific sample is the concentration that would be calculated if that analyte had a GC/MS response area equal to its area in the lowest level calibration standard used in the calibration. When an analyte is not detected in a sample or it has a response area that is smaller than its area in the lowest level calibration standard used, the concentration of the analyte in that sample is reported to be less than the value of its lower LOQ. When a specific analyte in a particular sample has a GC/MS response area that is larger than its area in the highest level calibration standard used in the calibration, the analyte amount is calculated using the relative response factor of that analyte in the highest level calibration standard used; the concentration is footnoted as exceeding the calibration range and is therefore an estimate.

For the HPLC/PDA method, the lower LOQ for a given analyte in a sample is the concentration that would be calculated if the analyte had a PDA response area equal to the minimum area needed to positively identify that analyte using the PDA spectral library. When an analyte is not detected in a sample or it has an area that is smaller than its minimum area, the concentration of the analyte in that sample is reported to be less than the value of its lower LOQ.

For the EA/IRMS method, the delta $(\delta)^{13}C$ and $\delta^{15}N$ values can be affected if the signals for the CO_2 and N_2 peaks are too small or too large. Sample results are not reported and the samples are reanalyzed if peak amplitudes exceed the following limits: for N_2, the mass 28 peak must have an amplitude of less than 9,500 millivolts (mV) and the mass 29 peak must have an amplitude greater than 500 mV; for CO_2, the mass 46 peak must have an amplitude of less than 9,500 mV and the mass 44 peak must have an amplitude greater than 500 mV. If peak amplitudes are near their limits, the accuracy of the result is less certain. Sample results are

footnoted to be used with caution if the peak amplitudes are as follows: for N_2, the mass 28 peak amplitude is between 7,500 and 9,500 mV or the mass 29 peak amplitude is between 500 and 750 mV; for CO_2, the mass 46 peak amplitude is between 7,500 and 9,500 mV or the mass 44 peak amplitude is between 500 and 750 mV.

For the TLC/FID method, a linear relationship between the amount of lipid spotted on a Chromarod and the FID response is needed for accurate lipid quantitation. Suitable ranges of the lipid amounts have been determined to be as follows: 0.30–7.5 microgram (µg) for sterol/wax esters, 0.30–7.5 µg for triglycerides, 0.25–10 µg for free fatty acids, 0.050–0.70 µg for cholesterol/sterols, and 0.050–1.0 µg for phospholipids/other polar lipids. In order for the amount of lipid to fall within the linear range of each lipid class, the volumes of the extracts are routinely adjusted as necessary, depending on the matrix, by diluting or concentrating the extracts and then reanalyzing the sample. If a lipid class is not detected in a sample (i.e., no signal above the baseline), a value of zero is reported for the proportion of that lipid class.

For the semiquantitative HPLC/fluorescence method, the amplitude of each analyte peak must remain on scale. If a peak amplitude goes "off scale" (i.e., recognized when the top of the peak is flat), the bile sample is diluted and reanalyzed. If an analyte peak is not detected in a sample (i.e., no signal above baseline), a value of zero is reported.

6.5. Quality Assurance Criteria for the Analytical Measurements

QA elements (e.g., subsections 6.5.1, Calibration, below, and 6.5.5, Method [Reagent] Blanks, page 22) are included in the analyses of every batch of samples. Acceptance criteria and required minimum frequency of analysis for each QA element are summarized in Tables 8–13. The results for the various QA elements are reviewed by laboratory personnel immediately following the analysis of each sample batch. These results are then used to determine when acceptance criteria have not been met and which corrective actions are required before analyses may proceed.

6.5.1. Calibration

Calibration for all methods is established before or during sample analyses and documentation is archived with the sample data. The GC/MS and TLC/FID methods require at least four concentration levels of calibration standards for analyte quantitation. The HPLC/PDA method uses at least four concentration levels of calibration standards to demonstrate the linearity of the detector's response and a single level for analyte quantitation. The EA/IRMS method employs at least two points for analyte ratio calibration. The HPLC/fluorescence method uses a single calibration point based on the average of the responses for repeated analyses of the calibration standard.

6.5.2. Continuing Calibration Verification

Continuing calibration verification (CCV) standards will be analyzed at the specified frequency, including at the beginning and end of every batch of samples. If CCV results do not meet specified criteria, then the entire batch and calibration standards must be reanalyzed. For the GC/MS and HPLC/PDA methods, the CCV standards' RSD of the analyte responses relative to the internal standard must be less than or equal to 15% for the repetitions. For the EA/IRMS

Table 8. Minimum analytical quality assurance criteria: Polycyclic aromatic compounds (PACs) and persistent organic pollutants (POPs) by gas chromatography/mass spectrometry (GC/MS).

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Once every batch of samples or once every two batches in one continuous analytical sequence	Analyte concentrations are to be calculated using point-to-point calibration with at least four concentration levels of calibration standards.
Continuing calibration	At start and end of every analytical sequence and every 10 or fewer field samples	The RSD of the analyte responses relative to the internal standard is to be ≤ 15% for the repetitions.
Reference materials: Sediment: NIST SRM 1944, NIST SRM 1941b Mussel tissue: NIST SRM 1974b Blubber: NIST SRM 1945 Fish tissue: NIST SRM 1946, NIST SRM 1947	One with every batch of 20 or fewer field samples	Concentrations of ≥ 70% of individual analytes are to be within 30% of either end of the 95% confidence interval of the reference values. These criteria do not apply to analytes with concentrations below their lower LOQ when the lower LOQ is within or greater than the 95% confidence interval, nor to those analytes known to have coeluting compounds.
Method blank	One with every batch of 20 or fewer field samples	No more than 5 analytes in a method blank are to exceed 2 × lower LOQ. Samples are not corrected for analytes found in the blank.
Sample replicates (i.e., duplicates or triplicates)	One with every 20 or fewer field samples	RSDs are to be ≤ 15% (equivalent to relative percent difference ≤ 30% for duplicates) for ≥ 90% of the analytes that have concentrations ≥ 1 ng/g.
Internal standards/surrogates	At least one internal standard/ surrogate is added to every sample	The recoveries are to be 60–130%.
Interlaboratory comparisons	At least one per year	In conjunction with the NIST or the IAEA.

Table 9. Minimum analytical quality assurance criteria: POPs by high-performance liquid chromatography/photodiode array (HPLC/PDA).

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Initial instrument set up	At least a four-point curve is to be used for instrument calibration. Standard curve correlation r ≥ 0.9900 for all analytes. Analyte concentrations are to be calculated using response factors from a single calibration solution.
Continuing calibration	At start and end of every analytical sequence (up to 14 instrument analyses)	The RSDs of the analyte responses relative to the internal standard are to be ≤ 15% for the repetitions. When the relative response factor for each analyte in the calibration standard in the current batch is compared to those of the same level standard for the original multilevel standard curve determination, the percent difference is to be ≤ 15% or the multilevel calibration will be repeated.
Reference materials: Sediment: NIST SRM 1944, NIST SRM 1941a, matrix spikes if needed Mussel tissue: NIST SRM 1974b Blubber: NIST SRM 1945 Fish tissue: NIST SRM 1946, NIST SRM 1947	One with every batch of 10–12 field samples	Concentrations of ≥ 70% of individual analytes are to be within 35% of either end of the 95% confidence interval range of the reference values. These criteria do not apply to analytes with concentrations < 10 × the lower LOQ or to those analytes known to have coeluting compounds.
Method blank	One with every batch of 10–-12 field samples	No more than 4 analytes in a method blank are to exceed 4 × lower LOQ, unless analyte is not detected in associated sample(s). Samples are not corrected for the analytes found in the method blank.
Sample replicates	One with every 20 field samples	RSDs are to be ≤ 30% (equivalent to relative percent difference ≤ 60% for duplicates) for ≥ 80% of analytes detected.
Internal standards/surrogates	Every sample	The recoveries are to be 60–120%.

Table 10. Minimum analytical quality assurance criteria: Fatty acids by gas chromatography/mass spectrometry (GC/MS).

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Once every batch of samples or once every two batches in one continuous analytical sequence	Analyte concentrations are to be calculated using point-to-point calibration with at least eight concentration levels of calibration standards.
Continuing calibration	At start and end of every analytical sequence and every 10 or fewer field samples	The RSD of the analyte responses relative to the internal standard are to be ≤ 15% for the repetitions.
Reference materials: Fish tissue: NIST SRM 1946, NIST SRM 1947 Blubber: NIST 1945	One with every batch of 24 or fewer field samples	Concentrations ≥ 70% of individual fatty acids are to be within 15% of either end of the 95% confidence interval range of the reference values. These criteria do not apply to analytes with concentrations below their lower LOQ when the lower LOQ is within or greater than the 95% confidence interval, nor to tentatively identified fatty acids, nor to those fatty acids known to have coeluting compounds (Table 4).
Method blank	One with every batch of 24 or fewer field samples	No more than five analytes in a method blank are to exceed 2 × lower LOQ.
Sample replicates (i.e., duplicates or triplicates)	One with every 24 or fewer field samples	RSDs are to be ≤ 15% (equivalent to relative percent difference ≤ 30% for duplicates) for ≥ 90% of the analytes.
Internal standards/surrogates	Every sample	The recoveries are to be 60–130%.
Interlaboratory comparisons	Infrequent and informal intercomparisons are available at present	In conjunction with the NIST or the IAEA, or through informal participation with government or university laboratories.

Table 11. Minimum analytical quality assurance criteria: Stable isotope ratios by elemental analyzer/isotope ratio mass spectrometry (EA/IRMS).

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Once every analytical sequence	At least a two-point line is to be used for instrument calibration and to calculate $\delta^{15}N$ and $\delta^{13}C$. A standard curve correlation of r > 0.9900 for $\delta^{15}N$ and $\delta^{13}C$.
Continuing calibration	At start and end of every analytical sequence and between every 10 field samples	Working standards: $\delta^{15}N$ values are to have a standard deviation $\leq \pm 0.30$ per mil and $\delta^{13}C$ values must have a standard deviation $\leq \pm 0.20$ per mil after the outliers have been removed. Outliers cannot exceed 20% of the total number of standards.
Reference material: Fish tissue: NIST SRM 1946	Between every 20 or fewer field samples with a minimum of four per analytical sequence	SRM: $\delta^{15}N$ and $\delta^{13}C$ values are to be within ± 0.30 and ± 0.20 per mil respectively of the interim value and the standard deviation must be ≤ 0.30 for $\delta^{15}N$ and ≤ 0.20 for $\delta^{13}C$. The Wt %N and Wt %C values are to be within $\pm 2.5\%$ and $\pm 5.0\%$ respectively of the calculated value or Wt %N or Wt %C values should not be reported.
Method blank	Three with every analytical sequence	Nitrogen peak amplitude and carbon dioxide peak amplitude are to be < 15% of the corresponding sample peak amplitude for two out of three of the blanks.
Maximum and minimum N_2 peak amplitudes	Each sample must have N_2 peak amplitudes between limit values	For N_2 the mass 28 peak is to have an amplitude of < 9,500 mV; if the amplitude is between 7,500 and 9,500 mV, it is to be footnoted to be used with caution. Also the mass 29 peak is to have an amplitude > 500 mV; if the amplitude is between 500 and 750 mV, it is to be footnoted to be used with caution.
Maximum and minimum CO_2 peak amplitudes	Each sample must have CO_2 peak amplitudes between limit values	For CO_2 the mass 46 peak is to have an amplitude of < 9,500 mV; if the amplitude is between 7,500 and 9,500 mV, it is to be footnoted to be used with caution. Also the mass 44 peak is to have an amplitude > 500 mV; if the amplitude is between 500 and 750 mV, it is to be footnoted to be used with caution.

Table 12. Minimum analytical quality assurance criteria: Percent lipid and lipid class proportions by thin-layer chromatography/flame ionization detection (TLC/FID).

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Every two weeks	At least a four-point line for each lipid class is to be used for instrument calibration. Concentrations of sterol/wax esters, triglycerides, free fatty acids, cholesterol/sterols and phospholipids/other polar lipids are to be calculated from each calibration. The standard curve is to have a correlation (r^2) > 0.98 for each of the five lipid classes.
Continuing calibration	One standard between every three field samples	The measured values for the CCV standards are to be ± 15% of the expected values.
Reference materials: Mussel tissue: NIST SRM 1974b Blubber: NIST SRM 1945 Fish tissue: NIST SRM 1946, NIST SRM 1947	For every 20 or fewer field samples with a minimum of four per batch	The percent lipid values of each NIST SRM are to be within 35% of either end of the 95% confidence interval of the reference value.
Method blank	One extraction method blank with every batch of 10–14 field samples and a solvent method blank between every three field samples	No peak is to be detected in a method blank or solvent blank.
Sample replicates	One with every 28 or fewer field samples	RSDs are to be ≤ 25% (equivalent to relative percent difference ≤ 50% for duplicates).
Interlaboratory comparisons	Informal and infrequent at present.	As defined by the NIST or through informal participation with comparable government or university laboratories or both.

Table 13. Minimum analytical quality assurance criteria: Metabolites of PACs by high-performance liquid chromatography (HPLC)/fluorescence.

Quality assurance element	Minimum frequency	Acceptance criteria
Instrument calibration	Once per analytical sequence	None.
Continuing calibration	At least three standards at the start of every analytical sequence, one every 15 field samples and at least one at the end of the analytical sequence	The RSD of the analyte responses relative to the internal standard is to be ≤ 10% for the repetitions.
Reference material: ASMBC2	One before and after the sample batch	Concentration of PAC equivalents are to be within the upper and lower control limits. These limits are defined as plus or minus two standard deviations of historical values for the control material.
Method blank	One HPLC method blank before and after each sample batch	PAC equivalents in the blank are to be less than 10% of the lowest concentration in any field sample in the sample batch.
Sample replicates	One with every 20 or fewer field samples	RSDs are to be ≤ 30% (equivalent to relative percent difference ≤ 60% for duplicates).

method, $\delta^{15}N$ values of the working stands must have a standard deviation less than or equal to plus or minus 0.30 per mil and $\delta^{13}C$ values of the working standards must have a standard deviation less than or equal to plus or minus 0.20 per mil after the statistically significant outliers have been removed. Outliers cannot exceed 20% of the total number of standards. For the TLC/FID method, the measured values for the CCV standards must be plus or minus 15% of the expected values. For the HPLC/fluorescence method, the RSD of the responses for the standard must be less than or equal to 10% for the repetitions.

6.5.3. Reference Materials

At least one SRM from the NIST, if available, is analyzed with every batch of field samples for quality assurance, except for analyses for PAC metabolites in bile, which uses an in-house control material (CM) (e.g., bile of Atlantic salmon exposed to 25 µg/mL of Monterey crude oil for 48 hours). The data resulting from the analyses of SRMs/CMs are reported in the same manner as field samples and are used to document the estimated accuracy of the associated field sample data. If the SRM results exceed the control limit criteria, then the entire batch of samples is to be considered suspect. The source of the error must be identified and corrected, and the samples may need to be reanalyzed, depending on the project requirements.

The laboratory's performance for PACs or POPs by the GC/MS method is considered acceptable if greater than or equal to 70% of reported values are within their control limits:

- Upper control limit = [1.3 × (certified concentration + uncertainty value for 95% confidence)].

- Lower control limit = [0.7 × (certified concentration – uncertainty value for 95% confidence)].

The laboratory's performance for fatty acids by the GC/MS method is considered acceptable if greater than or equal to 70% of reported values are within their control limits:

- Upper control limit = [1.15 × (certified concentration + uncertainty value for 95% confidence)].

- Lower control limit = [0.85 × (certified concentration – uncertainty value for 95% confidence)].

For all GC/MS methods, acceptance criteria do not apply to analytes that:

1) have a lower LOQ that is above the lower control limit,

2) that coelute with other compounds, or

3) do not have calibration standards available.

The laboratory's performance for POPs by the HPLC/PDA method is considered acceptable if greater than or equal to 70% of reported values are within their control limits:

- Upper control limit = [1.35 × (certified concentration + uncertainty value for 95% confidence)].

- Lower control limit = [0.65 × (certified concentration – uncertainty value for 95% confidence)].

These criteria apply only to those congeners that are greater than 10 times the lower LOQ and those that are free from coeluting substances.

There are no tissue SRMs certified for ratios of stable isotopes of carbon and nitrogen. An interim reference material (IRM) being used is a NIST SRM tissue (certified for POPs) which has been measured repeatedly in-house against IAEA primary standard materials. The laboratory's performance for the EA/IRMS method is considered acceptable if the mean of reported values is within plus or minus 0.30 per mil of the reference value for δ^{15}N, the mean of reported values is within plus or minus 0.20 per mil of the reference value for δ^{13}C, the standard deviation for δ^{15}N is less than or equal to 0.30 per mil and the standard deviation for δ^{13}C is less than or equal to 0.20 per mil. The Wt %N and Wt %C values should be within plus or minus 2.5% and plus or minus 5.0% respectively of the established mean IRM reference values.

For lipids, the percent lipid of each NIST SRM should be within its control limits:

- Upper control limit = [1.35 × (certified concentration + uncertainty value for 95% confidence)]

- Lower control limit = [0.65 × (certified concentration – uncertainty value for 95% confidence)]

There are no tissue SRMs certified for lipid classes.

For metabolites of PACs, an in-house bile control material is used because there are no bile SRMs. The laboratory's performance is considered acceptable if the reported values are within their control limits, defined as plus or minus two standard deviations from the mean of historical values for the control material.

6.5.4. Surrogate (Internal Standard) Recovery

All samples analyzed for PACs, POPs and fatty acids will be spiked with appropriate extraction surrogates (internal standards) as described in the laboratory SOPs. If a percent recovery does not meet the specified criteria, the sample will be reextracted and reanalyzed if possible (i.e., the sample is still available); otherwise the corresponding data will be qualified as being an estimate. For the GC/MS analyses, the measured percent recovery of the surrogates must be 60–130%; for the HPLC/PDA analyses, the measured percent recovery of the surrogates must be 60–120%.

6.5.5. Method (Reagent) Blanks

Method blanks are laboratory-derived samples that are subjected to the same analytical protocols as are the field samples. Failure to meet acceptance criteria requires definitive corrective action to identify and eliminate the source(s) of contamination before the subsequent reextraction or reanalysis of the batch of samples or both.

For the GC/MS methods, no more than five analytes in a method blank may exceed two times the value of the lower LOQ. For the HPLC/PDA method, no more than four analytes in a method blank may exceed four times the value of the lower LOQ, unless the analyte is not detected in any of the field samples in the batch.

For the EA/IRMS analyses, the method blanks are tin cups with no sample added, which are analyzed in the same manner as the environmental samples, but are not a measure of contamination that occurred during sample processing. These blanks are used to correct the $\delta^{15}N$ and $\delta^{13}C$ sample values for traces of nitrogen or carbon materials in the tin cups. The nitrogen and carbon dioxide peak amplitudes must be less than 15% of the corresponding sample peak amplitude for two out of three of the blanks or the samples must be reanalyzed after the problem has been corrected.

For the TLC/FID method, no peaks may be detected in a method blank. For the HPLC/fluorescence method, PAC equivalents in the blank must be less than 10% of the lowest concentration in any field sample in the analytical sample batch.

6.5.6. Sample Replicates

Field samples will be analyzed in replicate at the specified frequency to help ascertain whether samples are analytically homogeneous and to indicate whether other problems with reproducibility occurred during analysis. The reproducibility of the SRM results can indicate the precision for all analyses in the project.

For GC/MS analyses, replicate (i.e., duplicate or triplicate) samples are analyzed with approximately every 20 field samples, as sample amounts allow. RSDs are to be less than or equal to 15% (equivalent to relative percent difference $\leq$ 30% for duplicates) for greater than or equal to 90% of the analytes. For PACs and POPs, this applies only to those analytes that have concentrations greater than or equal to 1 ng/g.

For HPLC/PDA and HPLC/fluorescence analyses, duplicate samples will be analyzed approximately every 20 field samples. The RSDs are to be less than or equal to 30% (relative percent difference $\leq$ 60% for duplicates) for greater than or equal to 80% of analytes detected.

For EA/IRMS analyses, replicate samples of the SRM are analyzed every 20 or fewer field samples, with a minimum of four per analytical sequence, to show the reproducibility of the IRMS instrument. The standard deviations must be less than or equal to 0.30 for $\delta^{15}N$ and less than or equal to 0.20 for $\delta^{13}C$. Duplicate or triplicate field samples are suggested for approximately every 10 field samples, but are not used for QA. There are no acceptance criteria for replicate field samples because many explanations exist for widely varying values (e.g., problems with the sample processing or the homogeneity of the starting sample) that are often outside the control of the analytical laboratory. However, within-sample variability of the results for replicate field samples may be useful to the researcher.

For lipid classes by TLC/FID, duplicate samples will be analyzed at every 28 or fewer field samples. The RSDs are to be less than or equal to 25% (relative percent difference $\leq$ 50% for duplicates).

6.6. Laboratory Qualification of Data

Sample results that did not meet the quality assurance acceptance criteria or that presented analytical difficulties are footnoted by the laboratory so that the data user is aware of

the potential limitations of the data. These footnotes are summarized and presented in text (e.g., a report or case narrative) accompanying the data.

7.0. Data Reduction

Data reduction is the process whereby raw data (analytical measurements) are converted or reduced to usable results that are reported in the format specified for the project, including QA data. Primary data reduction is the responsibility of the analyst(s) conducting the analytical measurements, and is subject to further review by laboratory staff, the project leader, and the project manager(s).

Primary data reduction requires accounting for specific sample preparations, sample volume or weight analyzed, and any concentrations or dilutions required. All data reduction procedures are described in the laboratory's SOPs.

7.1. Reported Results

In general, results are reported as follows:

- To two (or, if requested, three) significant figures for PACs, POPs and PAC metabolites, and to three significant figures for fatty acids; to two figures following the decimal point for $\delta^{15}N$ and $\delta^{13}C$ values; and to one figure following the decimal point for Wt %N, Wt %C, C/N ratios, lipid classes, and percent lipid (by TLC/FID).

- For GC/MS and HPLC/PDA analyses, the analyte concentrations are calculated based on the surrogate compounds spiked into the sample prior to extraction.

- For GC/MS and HPLC/PDA analyses, percent recovery of surrogate standards are reported.

- For GC/MS and HPLC/PDA analyses for PACs and POPs, analytes in sediments are reported as ng/g dry weight and in tissues as ng/g wet weight. Percent dry weights of samples are determined for tissue analyses if requested.

- For GC/MS analyses for fatty acids, analytes are reported in units of fatty acid methyl esters as mass fraction (%) on a wet-mass basis.

- Results for analytes in method blanks are reported on the same basis as those for the samples being analyzed. The average of the sample weights for the field samples comprising a batch, as well as the average of the percent dry weights for sediments, is used in the calculations for the method blank.

- For PACs, POPs, and fatty acids, the lower LOQ is reported instead of a concentration when an analyte is below the quantitation range.

- If a lipid class is not detected in a sample, a value of zero is reported for the proportion of that lipid class.

- If a PAC metabolite peak is not detected by fluorescence in a sample, a value of zero is reported for that PAC equivalent.

- Data generated from the analysis of blank samples are not used for correction of analyte concentrations in samples, except for the EA/IRMS analyses.

- Replicate sample data is summarized as the mean plus or minus RSD.

7.2. Data Review

Data review is an internal process during which the data are reviewed and evaluated by laboratory personnel. This review is undertaken by analysts who are responsible for ensuring that the analytical data are correct and complete, the appropriate SOPs have been followed, and the QA results meet the acceptance criteria. It is the project manager's responsibility to ensure that all analyses performed by the laboratory are correct, complete, and meet project DQOs. The project leader has final review authority.

7.3. Laboratory Data Deliverables

The laboratory reports any difficulties encountered during sample preparation and analysis, as well as any limitations to the use of the data. In addition, the following specific information will be provided as requested:

- COC/sample receipt checklist,

- procedure modifications,

- calibration summaries (initial calibration data, correlation coefficients for HPLC/PDA method, continuing calibration data),

- data tables for field samples,

- QA data (surrogate recoveries, method blanks, SRMs, replicate field samples), as applicable, and

- corrective actions that were necessary.

8.0. Corrective Action/Procedure Alteration

The laboratory is required to adhere to the SOPs specified for the project unless procedure alterations are necessary to correct unforeseen analytical problems. Laboratory personnel are alerted that corrective action is necessary when QA data do not meet the acceptance criteria.

Because most of the corrective actions are handled at the laboratory level, it is the immediate responsibility of the analyst to identify and correct the situation before continuing with sample analysis. If the problem persists or cannot be identified, the matter is referred to the project leader or project manager for further investigation. Once resolved, a narrative describing 1) the problem, 2) the steps taken to identify and correct the problem, and 3) the action taken to remedy the problem in the relevant sample batches, must be prepared and submitted with the relevant data package. If the action involves a change from the accepted SOP, the SOP must be revised as appropriate.

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