United States Environmental Protection Agency | Department of the Army U.S. Army Corps of Engineers | EPA 823-B-95-001 April 1995

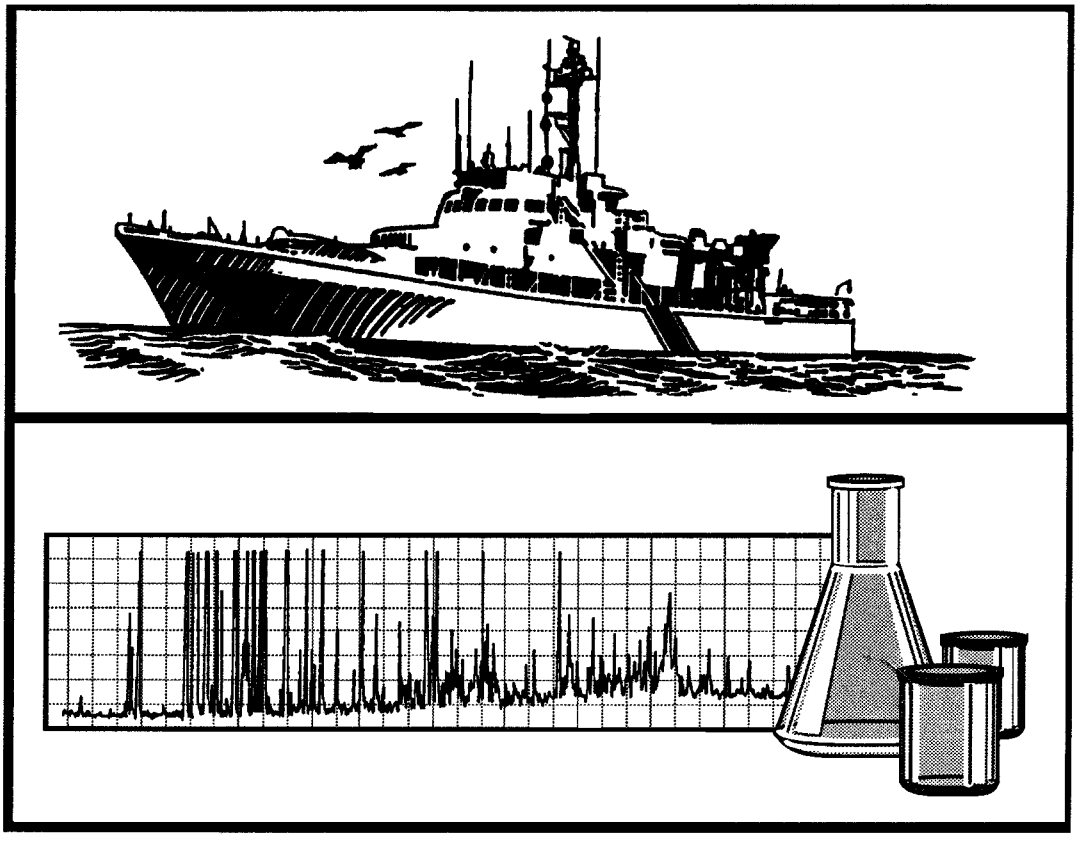
Office of Water (4305)

EPA

U.S. Army Corps of Engineers

QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations

Chemical Evaluations



QA/QC GUIDANCE FOR SAMPLING AND ANALYSIS OF SEDIMENTS, WATER, AND TISSUES FOR DREDGED MATERIAL EVALUATIONS

CHEMICAL EVALUATIONS

Prepared by

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and

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United States Army Corps of Engineers
Washington, D.C.

April 1995

The polices set out in this document are not final agency action, but are intended solely as guidance. They are not intended, nor can they be relied upon, to create any rights enforceable by any party in litigation with the United States. EPA officials may decide to follow the guidance provided in this document, or to act at variance with the guidance, based on an analysis of specific site circumstances. The Agency also reserves the right to change this guidance at any time without public notice.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

EPA 823-B-95-001

APR 28 1995

OFFICE OF WATER

Dear Colleagues:

The U.S. Environmental Protection Agency (EPA) is pleased to transmit a copy of the document titled *QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations. Chemical Evaluations*. This document was prepared in response to regional requests for quality assurance/quality control (QA/QC) guidance associated with the testing and evaluation of proposed dredged material discharges into inland or ocean waters. The workgroup that developed this national guidance was comprised of individuals from headquarters, field offices, and research laboratories of EPA and the U.S. Army Corps of Engineers (USACE) with experience related to dredged material discharge activities.

EPA and USACE technical guidance for evaluating the potential for contaminant-related impacts associated with the discharge of dredged material into inland and ocean waters, respectively, is found in the documents "Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.—Testing Manual (Draft)" (the *Inland Testing Manual*) (U.S. EPA and USACE 1994), and "Evaluation of Dredged Material Proposed for Ocean Disposal—Testing Manual" (the *Ocean Testing Manual*) (U.S. EPA and USACE 1991). Results of tests conducted using the testing manuals are the basis of independent evaluations made by EPA and USACE regarding the suitability of proposed dredged material for aquatic disposal.

This QA/QC guidance document serves as a companion document to the *Inland* and *Ocean Testing* manuals. The purpose of this document is as follows: 1) to provide guidance on the development of quality assurance project plans for ensuring the reliability of data gathered to evaluate dredged material proposed for discharge under the Clean Water Act or the Marine Protection Research and Sanctuaries Act, 2) to outline procedures that should be followed when sampling and analyzing sediments, water, and tissues, and 3) to provide recommended target detection limits for chemicals of concern. This document pertains largely to physical and chemical evaluations. Though it is directed primarily toward the evaluation of dredged material for aquatic disposal, it may be useful in other areas of dredged material assessment and management as well (e.g., disposal site monitoring or evaluation of alternative disposal options). The audience for this document is Federal and State agency personnel and public with an interest in the evaluation and management of

dredged material. The information provided herein is for the purpose of guidance only and does not constitute a regulatory requirement.

Requests for copies of this document (EPA document number EPA 823-B-95-001) should be sent to U.S. Environmental Protection Agency, National Center for Environmental Publications and Information, 11029 Kenwood Road, Building 5, Cincinnati, Ohio 45242.

We appreciate your continued interest in EPA's activities related to impact assessment of potentially contaminated sediments.

Sincerely,

Tudor T. Davies
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Office of Science
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Enclosure

QA/QC GUIDANCE FOR SAMPLING AND ANALYSIS OF SEDIMENTS, WATER, AND TISSUES FOR DREDGED MATERIAL EVALUATIONS

CHEMICAL EVALUATIONS

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April 1995

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ACRONYMS AND ABBREVIATIONS

AVS	acid volatile sulfide
BCF	bioconcentration factor
CLP	Contract Laboratory Program
CVAA	cold vapor atomic absorption spectrometry
CWA	Clean Water Act
EPA	U.S. Environmental Protection Agency
GC	gas chromatography
GC/ECD	gas chromatography/electron capture detection
GC/MS	gas chromatography/mass spectrometry
GFAA	graphite furnace atomic absorption spectrometry
ICP	inductively coupled plasma-atomic emission spectrometry
MPRSA	Marine Pollution, Research, and Sanctuaries Act
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-*p*-dioxin
PCDF	polychlorinated dibenzofuran
QAMP	quality assurance management plan
QAPP	quality assurance project plan
QA/QC	quality assurance and quality control
SRM	standard reference material
TCDD	tetrachlorodibenzo-*p*-dioxin
TDL	target detection limit
TEF	toxicity equivalency factor
TOC	total organic carbon
USACE	U.S. Army Corps of Engineers

ACKNOWLEDGMENTS

The contributions made by many individuals are gratefully acknowledged. The work group was comprised of individuals from headquarters, field offices, and research laboratories of the U.S. Environmental Protection Agency (EPA) and the U.S. Army Corps of Engineers (USACE) with experience related to dredged material discharge activities.

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1. INTRODUCTION

This document provides programmatic and technical guidance on quality assurance and quality control (QA/QC) issues related to dredged material evaluations. The U.S. Army Corps of Engineers (USACE) and U.S. Environmental Protection Agency (EPA) share the Federal responsibility for regulating the discharge of dredged material under two major acts of Congress. The Clean Water Act (CWA) governs discharges of dredged material into "waters of the United States," including all waters landward of the baseline of the territorial sea. The Marine Protection, Research, and Sanctuaries Act (MPRSA) governs the transportation of dredged material seaward of the baseline (in ocean waters) for the purpose of disposal.

EPA and USACE technical guidance for evaluating the potential for contaminant-related impacts associated with the discharge of dredged material into inland and ocean waters, respectively, is found in the documents "Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.—Testing Manual (Draft)" (the *Inland Testing Manual*) (U.S. EPA and USACE 1994), and "Evaluation of Dredged Material Proposed for Ocean Disposal—Testing Manual" (the *Ocean Testing Manual*) (U.S. EPA and USACE 1991). Results of tests conducted using the testing manuals are the basis of independent evaluations made by EPA and USACE regarding the suitability of proposed dredged material for aquatic disposal.

This QA/QC guidance document serves as a companion document to the *Inland* and *Ocean Testing* manuals. The purpose of this document is as follows: 1) to provide guidance on the development of quality assurance project plans for ensuring the reliability of data gathered to evaluate dredged material proposed for discharge under the CWA or the MPRSA, 2) to outline procedures that should be followed when sampling and analyzing sediments, water, and tissues, and 3) to provide recommended target detection limits (TDLs) for chemicals of concern. This document pertains largely to physical and chemical evaluations. Though it is directed primarily toward the evaluation of dredged material for aquatic disposal, it may be useful in other areas of dredged material assessment and management as well (e.g., disposal site monitoring or evaluation of alternative disposal options).

QA/QC planning is necessary to ensure that the chemical and biological data generated during dredged material evaluations meet overall program and specific project needs. Establishing QA/QC procedures is fundamental to meeting project data quality criteria and to providing a basis for good decision-making. The EPA has developed a two-tiered quality management structure

that addresses QA concerns at both the organizational level and at the technical/project level. QA management plans (known as QAMPs) identify the mission and customers of the organization, document specific roles and responsibilities of top management and employees, outline the structure for effective communications, and define how measures of effectiveness will be established. The quality standards, goals, performance specifications, and the QA/QC activities necessary to achieve them, are incorporated into project-specific QA project plans (known as QAPPs).

QA activities provide a formalized system for evaluating the technical adequacy of sample collection and laboratory analysis activities. These QA activities begin before samples are collected and continue after laboratory analyses are completed, requiring ongoing coordination and oversight. The QA program summarized in this document integrates management and technical practices into a single system to provide environmental data that are sufficient, appropriate, and of known and documented quality for dredged material evaluation.

QA project plans (QAPPs) provide a detailed plan for the activities performed at each stage of the dredged material evaluation (including appropriate sampling and analysis procedures) and outline project-specific data quality objectives that should be achieved for field observations and measurements, physical analyses, laboratory chemical analyses, and biological tests. Data quality objectives should be defined prior to initiating a project and adhered to for the duration of the project to guarantee acquisition of reliable data. This is accomplished by integrating quality control (QC) into all facets of the project, including development of the study design, implementation of sample collection and analysis, and data evaluation. QC is the routine application of procedures for determining bias and precision. QC procedures include activities such as preparation of replicate samples, spiked samples, blanks; calibration and standardization; and sample custody and recordkeeping. Audits, reviews, and compilation of complete and thorough documentation are QA activities used to verify compliance with predefined QC procedures. Through periodic reporting, these QA activities provide a means for management to track project progress and milestones, performance of measurement systems, and data quality.

A complete QA/QC effort for a dredged material testing program has two major components: a QA program implemented by the responsible governmental agency (the data user), and QC programs implemented by sampling and laboratory personnel performing the tests (the data generators). QA programs are also implemented by each field contractor and each laboratory. Typically, all field and laboratory data generators agree to adhere to the QA/QC of the data user for the contracted project as specified in the project QAPP. USEPA (1987a) provides useful guidance and may be followed on all points that are not in conflict with the guidance in this document. The guidance provided in this

document also incorporates information contained in U.S. EPA (1984a, 1991d) and U.S. EPA and USACE (1991, 1994).

1.1 GOVERNMENT (DATA USER) PROGRAM

Because the data generated in a dredged material evaluation are used for regulatory purposes, it is important to have proper QA oversight. The USACE, working in conjunction with the appropriate EPA Region(s), should implement a QA program to ensure that all program elements and testing activities (including field and laboratory operations) in the dredged material evaluation comply with the procedures in the QA project plan or with other specified guidelines for the production of environmental data of known quality. This QA guidance document was designed with the assistance of programmatic and scientific expertise from both EPA and USACE. Other qualified sources of QA program management should be contacted as appropriate. Some specific QA considerations in contract laboratory selection are discussed by Sturgis (1990) and U.S. EPA (1991d).

The guidance in this document is intended to assist EPA and USACE dredged material managers in developing QA project plans to ensure that: 1) the data submitted with dredged material permit applications are of high quality, sufficient, and appropriate for determining if dredging and disposal should occur; and 2) the contract laboratories comply with QC specifications of the regulations and guidelines governing dredged material evaluations. This includes the development of an appropriate QA management plan.

1.2 CONTRACTOR (DATA GENERATOR) PROGRAM

Each office or laboratory participating in a dredged material evaluation is responsible for using procedures which assure that the accuracy (precision and bias), representativeness, comparability, and completeness of its data are known and documented. To ensure that this responsibility is met, each participating organization should have a project manager and a written QA management plan that describes, in specific terms, the management approach proposed to assure that each procedure under its direction complies with the criteria accepted by EPA and USACE. This plan should describe a QA policy, address the contents and application of specific QA project plans, specify training requirements, and include other elements recommended by EPA quality assurance management staff (e.g., management system reviews). All field measurements, sampling, and analytical components (physical, chemical, and biological) of the dredged material evaluation should be discussed.

For the completion of a dredged material testing project, the project manager of each participating organization should establish a well-structured QA program that ensures the following:

- Development, implementation, and administration of appropriate QA planning documents for each study
- Inclusion of routine QC procedures for assessing data quality in all field and laboratory standard operating procedures
- Performance of sufficiently detailed audits at intervals frequent enough to ensure conformance with approved QA project plans and standard operating procedures
- Periodic evaluation of QC procedures to improve the quality of QA project plans and standard operating procedures
- Implementation of appropriate corrective actions in a timely manner.

The guidance provided in this document is intended to assist the data generator with the production of high-quality data in the field and in the laboratory (i.e., the right type and quality of information is provided to EPA and USACE to make a decision about the suitability of dredged material for aquatic disposal with the specified degree of confidence).

2. DRAFTING A QUALITY ASSURANCE PROJECT PLAN

A formal strategy should always be developed to obtain sufficient and appropriate data of known quality for a specific dredged material testing program. When the sample collection and laboratory analysis effort is small, this strategy may be relatively straightforward. However, when the sample collection and laboratory analysis effort is significant, the assurance of data quality may require the formulation of a formal and often quite detailed QA project plan. The QA project plan is a planning and an operational document.

The QA project plan should be developed by the applicant or contractor for each dredged material evaluation, in accordance with this document. The QA project plan provides an overall plan and contains specific guidelines and procedures for the activities performed at each stage of the dredged material testing program, such as dredging site subdivision, sample collection, bioassessment procedures, chemical and physical analyses, data quality standards, data analysis, and reporting. In particular, the QA plan addresses required QC checks, performance and system audits, QA reports to management, corrective actions, and assessment of data accuracy (precision and bias)[1], representativeness, comparability, and completeness. The plan should address the quantity of data required to allow confident and justifiable conclusions and decisions.

The following information should be included in each QA project plan for dredged material evaluation unless a more abbreviated plan can be justified (see U.S. EPA 1989a):

- Introductory material, including title and signature pages, table of contents, and project description
- QA organization and responsibilities (the QA organization should be designed to operate with a degree of independence from the technical project organization to ensure appropriate oversight)

[1] Historically, "accuracy" and "precision" have often been defined as separate and distinct terms. In particular, accuracy has often been taken to be only a measure of how different a value is from the true value (i.e., bias). However, data that have poor precision (i.e., high variability) may only have low bias *on the average* (i.e., close agreement to the true value). Therefore, recent literature (e.g., Kirchmer 1988) has defined accuracy as both the precision and bias of the data. This definition of accuracy is used throughout this guidance document.

- QA objectives
- Standard Operating Procedures
- Sampling strategy and procedures
- Sample custody
- Calibration procedures and frequency
- Analytical procedures
- Data validation, reduction, and reporting
- Internal QC checks
- Performance and system audits
- Facilities
- Preventive maintenance
- Calculation of data quality indicators
- Corrective actions
- QA reports to management
- References.

The remaining sections of this document provide more specific information on each of these items.

2.1 INTRODUCTORY MATERIAL

The following sections should be included at the beginning of every QA project plan:

- Title and signature pages
- Table of contents
- Project description
- Certification.

The signature page should be signed and dated by those persons responsible for approving and implementing the QA project plan. The applicant's project manager's signature should be included even if other persons are primarily responsible for QA activities. The headings in the table of contents should match the headings in the QA project plan. A list of figures, list of tables, and list of appendices should be included in the table of contents.

The goals and objectives of the study project should be outlined in the project description. The project description should illustrate how the project will be designed to obtain the information needed to achieve those goals. Sufficient detail and information should be included for regulatory agency decision-making.

The QA project plan should include the following certification statement signed by a duly authorized representative of the permittee:

> *I certify under penalty of law that this document and all attachments were prepared under my direction or supervision. The information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.*

2.2 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

A clear delineation of the QA organization and line of authority is essential for the development, implementation, and administration of a QA program. The relationship of the QA personnel to the overall project team and their responsibilities for implementing the QA program are identified in this section. In addition, guidance is provided for developing statements of work that address the responsibilities of contract laboratories used in the project.

2.2.1 Staffing for Quality Assurance

Organizational charts or tables should be used in the QA project plan to describe the management structure, personnel responsibilities, and the interaction among functional units. Each QA task should be fully described and the responsible individual, their respective telephone number, and the associated organization named. Names of responsible individuals should be included for the sampling team, the analytical laboratory, the data evaluation, QA/QC effort in the laboratory, and the data analysis effort. An example of a QA organization flow diagram is provided in Appendix A.

The project manager has overall responsibility for assuring the quality of data generated for a project. In most projects, actual QA activities are performed independent of the project manager. However, the project manager does ensure the implementation of any corrective actions that are called for during sampling, analysis, or data assessment. The writing of a QA project plan can usually be accomplished by one person with assistance as needed from

technical specialists for details of methods or QC criteria. One person should also have primary responsibility for coordinating the oversight of all sampling activities, including completion of all documentation for samples sent to the laboratory. Coordinating laboratory interactions before and during sample analysis is also best performed by one person to avoid confusion. Subsequent interactions that may be necessary with the laboratory during a QA review of the data may involve the persons actually doing the review.

Additional QC tasks and responsibilities during sampling and analysis are often assigned to technicians who collect samples, record field data, and operate and maintain sampling and analytical equipment. These technicians perform a number of essential day-to-day activities, which include calibrating and servicing equipment, checking field measurements and laboratory results, and implementing modifications to field or laboratory procedures. These individuals should have training to perform these functions and follow established protocols and guidelines for each of these tasks.

Technical staff are responsible for the validity and integrity of the data produced. The QA staff should be responsible for ensuring that all personnel performing tasks related to data quality are appropriately qualified. Records of qualifications and training of personnel should be kept current for verification by internal QA personnel or by regulatory agency personnel.

Technical competence and experience of all contract laboratory staff should be demonstrated. Staff qualifications should be documented, and training should be provided by the laboratory to encourage staff to attain the highest levels of technical competence. Staff turnover can affect the ability of a laboratory to perform a particular analysis. The experience of current staff with projects of similar scope should be assessed during the laboratory selection process. Technical competence and other factors such as the laboratory setup (including quality and capacity of the available analytical equipment), past experience (e.g., analysis of appropriate QC check samples and review of quarterly performance evaluation analyses), or an upfront demonstration of performance can be used to influence the project manager's selection. The need to conduct a comprehensive evaluation of candidate laboratories will vary with the project and the familiarity with available laboratories.

2.2.2 Statements of Work

Statements of work are prepared for both field work and laboratory analysis. Data quality requirements and analytical methods need to be clearly and concisely communicated to either USACE personnel performing the analyses or to the laboratory selected by USACE's or the permit applicant's project manager. These specifications are best contained in a written laboratory contract. The main body of the contract should consist of general terms and

conditions common to any legal contract. A statement of work should be appended to the contract. The statement of work should be drafted and negotiated with the laboratory prior to the start of any analyses. The statement of work should be written in clear and concise terms, providing sufficient detail and references to approved protocols for each required procedure or method to eliminate any confusion about steps in the analysis. The statement of work should define all requirements for acceptable analyses, an important consideration even when working with a familiar laboratory, and all pertinent information on the price, timing, and necessary documentation of the analyses. All available information on the range of concentrations expected and any special characteristics of the samples to be analyzed should also be contained in the statement of work. A generic statement of work for the analysis of most chemicals in the most commonly analyzed sample matrices is provided in Appendix B, and is based on the following outline:

- A summary of analyses to be performed, including:
  - A list of all variables to be analyzed for in each sample or group of samples
  - A list of all methods and target detection limits (TDLs) (see discussion in Section 2.3.2) for physical and chemical analyses and a list of test protocols for biological toxicity tests
  - The total number of samples provided for analysis and the associated laboratory QC samples, the cost of each analysis, and the total cost of the analytical service requested for each sample matrix.
- Acceptable procedures for sample delivery and storage, including:
  - The method of delivery, schedule of delivery, and person responsible for notifying the laboratory of any changes in the schedule
  - Requirements for physical storage of samples, holding times (consistent with those specified in the QA project plan), chain-of-custody, and sample logbook procedures.
- Methods to be followed for processing and analyzing samples.
- QA/QC requirements, including the data quality objectives specified in the QA project plan and appropriate warning and control limits.
- A list of products to be delivered by the laboratory, specifying the maximum time that may elapse between the submittal of samples to the laboratory and the delivery of data reports to the agency,

organization, or industry requesting the analyses. Penalties for late delivery (and any incentives for early delivery) should be specified, as should any special requirements for supporting documentation and electronic data files. A checklist of the laboratory deliverables for analysis of organic compounds, pesticides, and polychlorinated biphenyls (PCBs) is presented in Table 1. A checklist of laboratory deliverables for analysis of metals is presented in Table 2.

- Progress notices (usually necessary only for large projects).
- Circumstances under which the laboratory should notify project personnel of problems, including, for example, when control limits or other performance criteria cannot be met, instrument malfunctions are suspected, or holding time limits have or will shortly expire.
- Written authorization for any deviations from the sampling and analysis plan should be obtained from EPA and USACE before the deviation occurs.
- Notice that scheduled and unannounced laboratory visits by the project manager or representative may be conducted.

The following additional information should also be provided in the laboratory statement of work:

- Requirements that each laboratory submit a QA manual for review and approval by the agency, organization, or industry requesting or funding the analysis. Each manual should contain a description of the laboratory organization and personnel, facilities and equipment, analytical methods, and procedures for sample custody, quality control, data handling, and results of previous laboratory audits.
- Conditions for rejection or non-analysis of samples and reanalysis of samples.
- Required storage time for records and samples prior to disposal.
- Terms for payments to the laboratory, including a requirement that the quality of data must be acceptable (pending the outcome of the QA review) before payment is made.

Including these elements in the statement of work helps to assure that responsibilities, data requirements, and expectations for performance are clear. A copy of the statement of work should be provided to the individual performing the data assessment to assist in the evaluation of data returned by the laboratory.

TABLE 1. CHECKLIST OF LABORATORY DELIVERABLES FOR THE ANALYSIS OF ORGANIC COMPOUNDS

☐	A cover letter discussing analytical problems (if any) and referencing or describing the procedures and instrumentation used.
☐	Tabulated results, including final dilution volume of sample extracts, sample size, wet-to-dry ratios for solid samples (if requested), concentrations of compounds of interest (reported in units identified to two significant figures unless otherwise justified), and equations used to perform calculations. Concentration units should be µg/kg (dry weight) for sediment, and µg/L for water, µg/kg (wet weight) for tissue. These results should be checked for accuracy and the report signed by the laboratory manager or designee.
☐	Target detection limits (see discussion in Section 2.3.2 of this document), instrument detection limits, and detection limits achieved for the samples.
☐	Original data quantification reports for each sample.
☐	Method blanks associated with each sample, quantifying all compounds of interest identified in these blanks.
☐	A calibration data summary reporting the calibration range used. For the analysis of semivolatile organic compounds analyzed by mass spectrometry, this summary should include spectra and quantification reports for decafluorotriphenylphosphine (DFTPP) or an appropriate substitute standard. For volatile organic compounds analyzed by mass spectrometry, the summary should include spectra and quantification reports for bromofluorobenzene (BFB) or an appropriate substitute standard.
☐	Recovery assessments and replicate sample summaries. Laboratories should report all surrogate spike recovery data for each sample, and a statement of the range of recoveries should be included in reports using these data.
☐	All data qualification codes assigned by the laboratory, their description, and explanations for all departures from the analytical protocols.

TABLE 1. (cont.)

Additional Deliverables for Volatile or Semivolatile Organic Compound Analyses[a]

- ☐ Tentatively identified compounds (if requested) and methods of quantification, along with the three library spectra that best match the spectra of the compound of interest (see Appendix C, Figure 1 for an example of a library spectrum).
- ☐ Reconstructed ion chromatograms for gas chromatography/mass spectrometry (GC/MS) analyses for each sample.
- ☐ Mass spectra of detected compounds for each sample.
- ☐ Internal standard area summary to show whether internal standard areas were stable.
- ☐ Gel permeation chromatography (GPC) chromatograms (for analyses of semivolatile compounds, if performed), recovery assessments, and replicate sample summaries. Laboratories should report all surrogate spike recovery data for each sample, and a statement of the range of recoveries should be included in reports using these data.

Additional Deliverables for Pesticide and Polychlorinated Biphenyl Analyses[a]

- ☐ Gas chromatography/electron capture detection (GC/ECD) chromatograms for quantification column and confirmation columns for each sample and for all standards analyzed.
- ☐ GPC chromatograms (if GPC was performed).
- ☐ An evaluation summary for 4,4'-DDT/endrin breakdown.
- ☐ A pesticide standard evaluation to summarize retention time shifts of internal standards or surrogate spike compounds.

[a] Many of the terms in this table are discussed more completely in Appendix C.

TABLE 2. CHECKLIST OF LABORATORY DELIVERABLES FOR THE ANALYSIS OF METALS

☐	A cover letter discussing analytical problems (if any) and referencing or describing the digestion procedures and instrumentation used.
☐	Tabulated results for final dilution volumes of sample digestates, sample size, wet-to-dry ratios for solid samples (if requested), and concentrations of metals (reported in units identified to two significant figures unless otherwise justified). Concentration units should be µg/kg (dry weight) for sediment, µg/L for water, and µg/kg (wet weight) for tissue.[a] These results should be checked for accuracy and the report signed by the laboratory manager or designee.
☐	Target detection limits (see discussion in Section 2.3.2 of this document), instrument detection limits, and detection limits achieved for the samples.
☐	Method blanks for each batch of samples.
☐	Results for all the quality control checks and initial and continuing calibration control checks conducted by the laboratory.
☐	All data quantification codes assigned by the laboratory, their description, and explanations for all departures from the accepted analytical protocols.

[a] Most laboratories will report metals data in mg/kg for solid samples. The specification here of µg/kg is for consistency with organic chemical analyses, which are typically reported as µg/kg for solid samples. If different units are used, care should be taken to ensure that results are not confused.

2.3 QUALITY ASSURANCE OBJECTIVES

Data quality objectives are addressed in this section of the QA project plan. Data quality objectives define performance-based goals for accuracy (precision and bias), representativeness, comparability, and completeness, as well as the required sensitivity of chemical measurements (i.e., TDLs). Accuracy is defined in terms of bias (how close the measured value is to the true value) and precision (how variable the measurements are when repeated) (see footnote at the beginning of Section 2). Data quality objectives for the dredged material program are based on the intended use of the data, technical feasibility, and consideration of cost. Therefore, data that meet all data quality objectives should be acceptable for unrestricted use in the project and should enable all project objectives to be addressed.

Numerical data quality objectives should be summarized in a table, with all data calculated and reported in units consistent with those used by other organizations reporting similar data, to allow comparability among databases. All measurements should be made so that results are representative of the medium (e.g., sediments, water, or tissue) being measured. Data quality objectives for precision and bias established for each measurement parameter should be based on prior knowledge of the measurement system employed, method validation studies, and the requirements of the specific project. Replicate tests should be performed for all test media (e.g., sediments, water, or tissue). Precision of approximately ≤ 30–50 relative percent difference between measurements (the random error of measurement) and bias of 50–150 percent of the true value (the systematic error of measurement) are adequate in many programs for making comparisons with regulatory limits. Precision may be calculated using three or more replicates to obtain the standard deviation and the derived confidence interval. Bias may be determined with standard reference material (SRM) or by spiking analyte-free samples.

These data quality objectives define the acceptability of laboratory measurements and should also include criteria for the maximum allowable time that samples or organisms can be held prior to analysis by a laboratory. An example of a data quality objectives summary for laboratory measurements is provided in Appendix A.

2.3.1 Program vs. Project Objectives

This document provides general guidance for QA activities conducted during dredged material evaluations. However, specific project needs will affect the kinds of chemical analyses that are requested by the project manager. Special project needs should be identified during preparation of the QA project plan and should be documented in this section of the plan. For example, a preliminary

reconnaissance of a large area may only require data from simple and quick checks performed in the field. In contrast, a complete characterization of contamination in a sensitive area may require specialized laboratory methods, lower TDLS, and considerable documentation of results.

Before defining the analyses that should be performed to meet the data quality objectives established on a project-specific basis, a thorough review of all historical data associated with the site (if applicable) should be performed (see discussions in U.S. EPA and USACE 1991, 1994). A review of the historical data should be conducted in response to data needs in the testing program. A comprehensive review of all historical data should eliminate unnecessary chemical analyses and assist in focusing the collection of chemical-specific data that are needed. A more thorough discussion of how to review and use historical data is provided in Section 2.5.2.

2.3.2 Target Detection Limits for Chemicals

Different analytical methods are capable of detecting different concentrations of a chemical in a sample. In general, as the sensitivity and overall accuracy of a technique increases, so does the cost. Recommended TDLs that are judged to be feasible by a variety of methods, cost effective, and to meet the requirements for dredged material evaluations are summarized in Table 3 (at the end of Section 2.4), along with example analytical methods that are capable of meeting the TDLs. However, any method that can achieve these TDLs is acceptable, provided that the appropriate documentation of the method performance is generated for the project.

The TDL is a performance goal set between the lowest, technically feasible, detection limit for routine analytical methods and available regulatory criteria or guidelines for evaluating dredged material (see summaries in McDonald et al. [1992]; PSEP [1991]). The TDL is, therefore, equal to or greater than the lowest amount of a chemical that can be reliably detected based on the variability of the blank response of routine analytical methods (see Section 2.10.1 for discussion of method blank response). However, the reliability of a chemical measurement generally increases as the concentration increases. Analytical costs may also be lower at higher detection limits. For these reliability, feasibility, and cost reasons, the TDLs in Table 3 have been set at not less than 10 times lower than available regional or international dredged material guidelines for potential biological effects associated with sediment chemical contamination. In many cases, lower detection limits than the TDL can be obtained and may be desired for some regional programs (e.g., for carefully documenting changes in conditions at a relatively pristine site).

All data generated for dredged material evaluation should meet the TDLs in Table 3 unless a regional requirement is made or sample-specific interferences

occur. Any sample-specific interferences should be well documented by the laboratory. If significantly higher or lower TDLs are required to meet rigorously defined data quality objectives (e.g., for human health risk assessments) for a specific project, then on a project-specific basis, modification to existing analytical procedures may be necessary. Such modifications should be documented in the QA project plan. An experienced analytical chemist should be consulted so the most appropriate method modifications can be assessed, the appropriate coordination with the analytical laboratory can be implemented, and the data quality objectives can be met. A more detailed discussion of method modifications is provided in Section 2.8.2.2.

2.4 *STANDARD OPERATING PROCEDURES*

Standard operating procedures are written descriptions of routine methods and should be provided for as many methods used during the dredged material evaluation as possible. A large number of field and laboratory operations can be standardized and presented as standard operating procedures. Once these procedures are specified, they can be referenced or provided in an appendix of the QA project plan. Only modifications to standard operating procedures or non-standard procedures need to be explained in the main body of the QA project plan (e.g, sampling or analytical procedures summaries discussed in Sections 2.5 and 2.8, respectively).

General types of procedures that benefit from standard operating procedures include field measurements ancillary to sample collection (e.g., depth of overlying water, sampling depth, water quality measurements or mixing model input measurements), chain-of-custody, sample handling and shipment, and routine analytical methods for chemical analyses. Standard operating procedures ensure that all persons conducting work are following the same procedures and that the procedures do not change over time. All personnel should be thoroughly familiar with the standard operating procedures before work is initiated. Deviations from standard operating procedures may affect data quality and integrity. If it is necessary to deviate from approved standard operating procedures, these deviations should be documented and approved through an appropriate chain-of-command. Personnel responsible for ensuring the standard operating procedures are adhered to should be identified in the QA project plan. Example standard operating procedures are provided in Appendix D.

TABLE 3. ROUTINE ANALYTICAL METHODS AND TARGET DETECTION LIMITS FOR SEDIMENT, WATER, AND TISSUE (parts per billion, unless otherwise noted)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Inorganic Chemicals						
Aluminum	3050A/6010A; U.S. EPA (1993a)	50,000[c]	200.8; U.S. EPA (1993a);	1,000	202.2; 6010A/200.7	40
Antimony	3050A; 7040/7041; U.S. EPA (1993a); PSEP (1990a)	2,500	200.8; 7040/7041; U.S. EPA (1993a);	100	7041; 204.2	3
Arsenic	7061; 7060A; 3050A; U.S. EPA (1993a); PSEP (1990a); EPRI (1986)	5,000	200.8/7061; 7060A; U.S. EPA (1993a)	100	3010; 7061; 206.2; 206.3; EPRI (1986)	1
Beryllium	200.8; 7090/7091; U.S. EPA (1993a)	2,500[d]	200.8; 7090/7091; U.S. EPA (1993a)	100	7091; 210.2; 6010A/200.7; 200.8	0.2
Cadmium	3050A; 6010A; 7131A/7130; U.S. EPA (1993a); PSEP (1990a)	300	200.8; 7131A; 7130; U.S. EPA (1993a)	100	213.2; 7131A; 3010; 6010A/200.7; 200.8	1
Chromium	3050A/7191; 7190; 6010A; U.S. EPA (1993a);	5,000	200.8/7191; 7190; U.S. EPA (1993a)	100	7191; 200.8; 218.2; 3010; 6010A/200.7	1

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Cobalt	7201	100	200.8; 7201	100	219.2	4
Copper	3050A/7211; 7210; 6010A; U.S. EPA (1993a); PSEP (1990a)	5,000[c]	200.8/7211; 7210; U.S. EPA (1993a)	100	7211; 200.8; 220.1; 220.2; 3010; 6010A/200.7	1
Hexavalent chromium	--[f]	--	--[f]	--	7197; 218.5	50
Iron	3050A/7381; U.S. EPA (1993a)	50,000[d]	200.8; 7381; 6010A; U.S. EPA (1993a)	10,000	6010A/200.7; 3010; 7381; 236.2	10
Lead	3050A/7421; 7420; 6010A; U.S. EPA (1993a); PSEP (1990a)	5,000	200.8/7421; 7420; U.S. EPA (1993a)	100	7421; 239.2	1
Manganese	3050A/7461; U.S. EPA (1993a)	5,000[c]	200.8/7461; U.S. EPA (1993a)	500	6010A/200.7; 243.2; 3010	1
Mercury	7471; U.S. EPA (1993a)	200	7471; U.S. EPA (1993a)	10	7471; 245.1; 245.2	0.2
Nickel	3050A/6010A; 7521; 7520; U.S. EPA (1993a); PSEP (1990a)	5,000	200.8/6010A; 7521; 7520; U.S. EPA (1993a)	100	6010A; 7521; 249.2	1
Selenium	7741; 7740; U.S. EPA (1993a); EPRI (1986)	1,000[c]	200.8/7741; 7740; U.S. EPA (1993a)	200	7740; 7741; 270.2; 270.3; EPRI (1986)	2

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Silver	3050A/7761; 7760; U.S. EPA (1993a); PSEP (1990a)	200[a]	200.8/7761; 7760; U.S. EPA (1993a)	100	7761; 272.2	1
Thallium	7840/7841; U.S. EPA (1993a)	200[c,e]	200.8; 7840; 7841; U.S. EPA (1993a)	100	7840; 7841; 279.2	1
Tin	U.S. EPA (1993a)	500[c,e]	200.8; U.S. EPA (1993a)	100	282.2	5
Zinc	3050A/7951; 7950; U.S. EPA (1993a); PSEP (1990a)	15,000	200.8/7951; 7950; U.S. EPA (1993a)	2,000	7951; 289.2; 200.7; 3010; 6010A	1
Organotin	NCASI (1986); Uhler and Durrel (1989); Rice et al. (1987)	10	NCASI (1986); Rice et al. (1987); Uhler et al. (1989)	10	NCASI (1986); Rice et al. (1987); Uhler and Durrel (1989)	0.01

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Nonionic Organic Compounds						
LPAH Compounds						
Naphthalene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; NOAA (1989); U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a) Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Acenaphthylene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Acenaphthene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a) Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Fluorene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Phenanthrene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Anthracene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
1-Methylnaphthalene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
2-Methylnaphthalene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
HPAH Compounds						
Fluoranthene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Pyrene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Benz[a]anthracene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10

T. : 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Chrysene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Benzo(b&k)fluoranthenes	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Benzo[a]pyrene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Indeno[1,2,3-c,d]pyrene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Dibenz[a,h]anthracene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Benzo[g,h,i]perylene	1625C; 3540A; 3550A/8100; 8250; 8270A; 8310; U.S. EPA (1993a)	20	1625C; 8100; 8250; 8270A; 8310; U.S. EPA (1993a); Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8250; 8270A; 8310	10
Chlorinated Benzenes						
1,3-Dichlorobenzene	1625C; 3540A; 3550A/8100; 8240A; 8250; 8260; 8270A	20	1625C; 8240A: 8250; 8270A; Sloan et al. (1993)	20	1625C; 3510A; 3520A/8100; 8240A; 8250; 8260; 8270A	10
1,4-Dichlorobenzene	1625C; 3540A; 3550A/8100; 8240A; 8250; 8260; 8270A	20	1625C: 8100; 8240A; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8240A; 8250; 8260; 8270A	10
1,2-Dichlorobenzene	1625C; 3540A; 3550A/8100; 8240A; 8250; 8260; 8270A	20	1625C; 8240A: 8250; 8270A; Sloan et al. (1993)	20	1625C; 3510A; 3520A/8100; 8240A; 8250; 8260; 8270A	10
1,2,4-Trichlorobenzene	1625C; 3540A; 3550A/8250; 8260; 8270A	10[f]	1625C; 8250; 8260; 8270A; Sloan et al. (1993)	20	1625C; 3510A; 3520A/8250; 8260; 8270A	10
Hexachlorobenzene	1625C; 3540A; 3550A/8250; 8260; 8270A	10[f]	1625C; 8250; 8260; 8270A; Sloan et al. (1993)	20	1625C; 3510A; 3520A/8250; 8260; 8270A	10

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Phthalate Esters						
Dimethyl phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8060; 8100; 8250; 8270A	10
Diethyl phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8060; 8250; 8270A	10
Di-n-butyl phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8060; 8250; 8270A	10
Butyl benzyl phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8060; 8250; 8270A	10
Bis[2-ethylhexyl]phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8060; 8250; 8270A	10

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Di-n-octyl phthalate	1625C; 3540A; 3550A/8060; 8100; 8250; 8270A	50	1625C; 8060; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8100; 8060; 8250; 8270A	10
Miscellaneous Extractable Compounds						
Benzyl alcohol	1625C; 3540A; 3550A/8250; 8270A	50	1625C; 8250; 8270A	100	1625C; 3510A; 3520A/8250; 8270A	50
Benzoic acid	1625C; 3540A; 3550A/8250; 8270A	100	1625C; 8250; 8270A	100	1625C; 3510A; 3520A/8250; 8270A	50
Dibenzofuran	1625C; 3540A; 3550A/8250; 8270A	50	1625C; 8100; 8250; 8270A; Sloan et al. (1993); NOAA (1989)	20	1625C; 3510A; 3520A/8250; 8270A	10
Hexachloroethane	1625C; 3540A; 3550A/8250; 8270A	100	1625C; 8250; 8270A	40	1625C; 3510A; 3520A/8250; 8270A	50
Hexachlorobutadiene	1625C; 3540A; 3550A/8250; 8270A	20	1625C; 8250; 8270A	40	1625C; 3510A; 3520A/8250; 8270A	50

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
N-Nitrosodiphenylamine	1625C; 3540A; 3550A/8250; 8270A	20	1625C; 8250; 8270A	20	1625C; 3510A; 3520A/8250; 8270A	50
Methylethyl ketone	1624C; 3540A; 3550A/8250; 8240A; 8260; 8270A	20	1624C; 8250; 8270A	20	1624C; 3510A; 3520A/8250; 8240A; 8260; 8270A	50
Polychlorinated Dibenzofurans						
Tetrachlorinated furans	1613; 8290	0.001	8290	0.001	1613; 8290	0.00001
Pentachlorinated furans	1613; 8290	0.0025	8290	0.0025	1613; 8290	0.000025
Hexachlorinated furans	1613; 8290	0.005	8290	0.005	1613; 8290	0.00005
Heptachlorinated furans	1613; 8290	0.005	8290	0.005	1613; 8290	0.00005
Octachlorinated furans	1613; 8290	0.01	8290	0.01	1613; 8290	0.0001
Polychlorinated Dibenzo-*p*-dioxins						
2,3,7,8-TCDD	1613; 8290	0.001	8290	0.001	1613; 8290	0.00001
Other tetrachlorinated dioxins	1613; 8290	0.001	8290	0.001	1613; 8290	0.00001
Pentachlorinated dioxins	1613; 8290	0.0025	8290	0.0025	1613; 8290	0.000025
Hexachlorinated dioxins	1613; 8290	0.005	8290	0.005	1613; 8290	0.00005

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Heptachlorinated dioxins	1613; 8290	0.005	8290	0.005	1613; 8290	0.00005
Octachlorinated dioxins	1613; 8290	0.01	8290	0.01	1613; 8290	0.0001
PCBs						
PCB congeners	Sloan et al. (1993); NOAA (1989); U.S. EPA (1993a)	1	NOAA (1989); U.S. EPA (1993a)	2	NOAA (1989)	0.01
Pesticides						
Aldrin	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.04
Chlordane and derivatives	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.14
Dieldrin	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.02
4,4'-DDD	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
4,4'-DDE	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
4,4'-DDT	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
Endosulfan and derivatives	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
Endrin and derivatives	3540A; 3550A/8080; NOAA (1985)	5	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
Heptachlor and derivatives	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
γ-Hexachlorocyclohexane (lindane)	3540A; 3550A/8080; NOAA (1985)	10	8080; NOAA (1985)	10	608; 3510A; 3520A/8080	0.1
Toxaphene	3540A; 3550A/8080	50	8080	50	608; 3510A; 3520A/8080	0.5
Methoxychlor	3540A; 3550A/8080	10	8080	10	608; 3510A; 3520A/8080	0.5
Chlorbenside	3540A; 3550A/8080; NOAA (1985)	2	8080; NOAA (1985)	2	608; 3510A; 3520A/8080	0.002

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Dacthal	3540A; 3550A/8080; NOAA (1985)	2	8080; NOAA (1985)	2	608; 3510A; 3520A/8080	0.03
Total chlorinated pesticides	3540A; 3550A/8080; NOAA (1985)	20	8080; NOAA (1985)	20	608; 3510A; 3520A/8080	0.02
Malathion	3540A; 3550A/8141	5	8141	5	3510A; 3520A/8141	0.8
Parathion	3540A; 3550A/8141	6	8141	6	3510A; 3520A/8141	0.8
Volatile Organic Compounds						
Benzene	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Chloroform	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Ethylbenzene	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Toluene	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Trichloroethene	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Tetrachloroethene	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Total xylenes	1624C; 8240A; 8260	10	1624C; 8240A; 8260	10	1624C; 8240A; 8260	5
Ionizable Organic Compounds						
Phenols						
Phenol	1625C; 3540A; 3550A/8040A; 8250; 8270A; 9066	100	1625C; 8040A; 8270A	20	1625C; 3510A; 3520A/8040A; 8250; 8270A	10
2-Methylphenol	1625C; 3540A; 3550A/8040A; 8250; 8270A	50	1625C; 8040A; 8270A	20	1625C; 3510A; 3520A/8040A; 8250; 8270A	10
4-Methylphenol	1625C; 3540A; 3550A/8040A; 8250; 8270A	100	1625C; 8040A; 8270A	20	1625C; 3510A; 3520A/8040A; 8250; 8270A	10
2,4-Dimethylphenol	1625C; 3540A; 3550A/8040A; 8250; 8270A	20	1625C; 8040A; 8270A	20	1625C; 3510A; 3520A/8040A; 8250; 8270A	10
Pentachlorophenol	1625C; 3540A; 3550A/8040A; 8250; 8270A	100	1625C; 8040A; 8270A	100	1625C; 3510A; 3520A/8040A; 8250; 8270A	50
Resin Acids and Gualacols	1625C; 3540A; 3550A; 8250; 8270A	10	--[g]	--	--	--

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Other Analyses						
Ammonia	350.1; 350.2; Plumb (1981)	100	--	--	350.1; 350.2; 350.3	30
Cyanides	9010A; 9012	2,000	9010A; 9012	1,000	335.2	5,000
Total organic carbon	PSEP (1986); U.S. EPA (1987a, 1992b)	0.1%	--	--	9060; 415.1; APHA 5310D	0.1%
Total petroleum hydrocarbons	9070; 418.1;	5,000	418.1	100,000	418.1	100
Total recoverable petroleum hydrocarbons	418.1	5,000	--	--	418.1	500
Total phenols	8040A	1,000	8040A	10,000	420.1; 625; 8040A	50
Acid-volatile sulfides	Cutter and Oates (1987); U.S. EPA (1991a); DiToro et al. (1990)	0.1µmole/g	--	--	--	--
Total sulfides	9030; Plumb (1981)	100	--	--	376.2	100
Grain size	Plumb (1981); ASTM (1992)	1%	--	--	--	--
Total suspended solids	--	--	--	--	160.2; APHA 2510D	1.0 mg/L
Total settleable solids	--	--	--	--	160.5; APHA 2540B	0.05 ml/L

TABLE 3. (cont.)

Chemical	Example Sediment Method[a]	Recommended Sediment TDL	Example Tissue Method[a]	Recommended Tissue TDL[b]	Example Water Method[a]	Recommended Water TDL[b]
Total solids/dry weight	160.3; Plumb (1981); PSEP (1986)	0.1%	--	--	--	--
Total volatile solids	160.4; Plumb (1981); APHA 2540E; PSEP (1986)	0.1%	--	--	--	--
Specific gravity	Plumb (1981)	0.01 mg/L	--	--	--	--
pH	9045; Plumb (1981)	0.1 pH units	--	--	Plumb (1981)	0.1 pH units
Total water content of test species	--	--	U.S. EPA (1986b, 1987a)	0.1%	--	--
Total lipid	--	--	Bligh and Dyer (1959); Folch et al. (1957)	0.1%	--	--

Note: HPAH - high molecular weight polycyclic aromatic hydrocarbon
LPAH - low molecular weight polycyclic aromatic hydrocarbon
TCDD - tetrachlorodibenzo-*p*-dioxin
TDL - The target detection limit is a performance goal set between the lowest, technically feasible, detection limit for routine analytical methods and available regulatory criteria or guidelines for evaluating dredged material. The target detection limit is, therefore, equal to or greater than the lowest amount of a chemical that can be reliably detected based on the variability of the blank response of routine analytical methods. However, the reliability of a chemical measurement generally increases as the concentration increases. Analytical costs may also be lower at higher detection limits. For these reasons, the target detection limit has been set not less than 10 times lower than available dredged material guidelines.

[a] Numbered methods are found in references as listed on following page.

[b] Determined by work group discussion; no or few effects guidelines are available for comparison.

TABLE 3. (cont.)

[c] No sediment screening or adverse effects guidelines are available for comparison.

[d] Less than 1/10 of available sediment guidelines for screening concentrations or potential adverse effects, but still cost effective and feasible to attain with a range of routine analytical methods.

[e] TDL may restrict use of some routine analytical methods, but reflects work group consensus.

[f] Sediment TDL slightly exceeds one available sediment guideline (Washington Sediment Management Standards) at low organic carbon content (< 2 percent TOC).

[g] -- - Not applicable.

REFERENCES CONTAINING NUMBERED METHODS IN TABLE 3.

Reference	Method				
US EPA 1983	160.2	206.2	220.2	270.2	350.1
	160.3	206.3	236.2	270.3	350.2
	160.4	210.2	239.2	272.2	350.3
	160.5	213.2	243.2	279.2	376.2
	200.7	218.2	245.1	282.2	415.1
	200.8	218.5	245.2	289.2	418.1
	202.2	219.2	249.2	335.2	420.1
	204.2	220.1			
US EPA 1982	608	625			
US EPA 1989b	8290				
US EPA 1990f	1613				
US EPA 1989c	1624C	1625C			
US EPA 1986a	3010	7090	7420	7841	8270A
	3050A	7091	7421	7950	8310
	3510A	7130	7461	7951	9010A
	3520A	7131A	7471	8040A	9012
	3540A	7190	7520	8060	9030
	3550A	7191	7521	8080	9045
	6010A	7197	7740	8100	9060
	7040	7201	7741	8141	9066
	7041	7210	7760	8240A	9070
	7060A	7211	7761	8250	
	7061	7381	7840	8260	
APHA 1989	APHA 2510D	APHA 2540B	APHA 2540E	APHA 5310D	

2.5 SAMPLING STRATEGY AND PROCEDURES

A sampling strategy should be developed to ensure that the sampling design supports the planned data use. For example, a project that was planned to characterize a specific area would have different sampling design requirements than a project that was screening for a suspected problem chemical. The sampling strategy will strongly affect the representativeness, comparability, and completeness that might be expected for field measurements. In addition, the strategy for collecting field QC samples (e.g., replicates) will assist in the determination of how well the total variability of a field measurement can be documented. Therefore, development of the sampling strategy should be closely coordinated with development of QA objectives discussed in Section 2.3.

Specific procedures for collecting each kind of sediment, water, tissue, or biological sample are described in this section of the QA project plan. The level of detail can range from a brief summary of sampling objectives, containers, special sample handling procedures (including compositing and subsampling procedures, if appropriate), and storage/sample preservation to a complete sampling plan that provides all details necessary to implement the field program. Standard operating procedures do not require elaboration in this section of the QA project plan (see Section 2.4).

If complete sampling details are not provided in the QA project plan, then reference should be made to the sampling plan that does provide all details. The QA project plan may be an appendix of the sampling plan, or specific sampling details may be provided as an appendix of the QA project plan. For smaller projects, a single planning document may be created that combines a work plan (project rationale and schedule for each task), detailed sampling plan (how project tasks are implemented), and the QA project plan. For larger projects, the QA project plan and the detailed sampling plan may be two separate documents.

This section of the document provides basic guidance for assuring sample quality from collection to delivery to the laboratory and guidance on items to consider when designing a sampling plan. A well-designed sampling plan is essential when evaluating the potential impact of dredged material discharge on the aquatic environment. The purpose of the sampling plan is to provide a blueprint for all fieldwork by defining in detail the appropriate sampling and data collection methods (in accordance with the established QA objectives; see Section 2.3). Before any sampling is initiated, the sampling plan should meet clearly defined objectives for individual dredging projects. Factors such as the availability and content of historical data, the degree of sediment heterogeneity, the volume of sediment proposed to be dredged, the areal extent of the dredging project, the number and geographical distribution of sample collection sites, potential contaminant sources, and the procedures for collection,

preservation, storage, and tracking of samples should be carefully considered and are necessary for adequate QA/QC of the data. The magnitude of the dredging project and its time and budgetary constraints should also be considered.

The following kinds of information should be reviewed for assistance in designing the sampling plan:

- **Geochemical and hydrodynamic data**—The grain size, specific density, water content, total organic carbon (TOC), and identification of sediment horizons are helpful in making operational decisions. Areas of high tidal currents and high wave energy tend to have sediments with larger grain sizes than do quiescent areas. Many contaminants have a greater affinity for clay and silt than for sand. Horizontal and vertical gradients may exist within the sediment. If the sediments are subject to frequent mixing by wave action, currents or prop wash, the sediments are likely to be relatively homogenous. Local groundwater quality and movement should be determined if groundwater is a potential source of contamination.

- **Quality and age of available data**—Reviewing the results of chemical analyses performed in past studies can help in selecting the appropriate contaminants of concern and in focusing plans for additional analyses. In particular, analytical costs can be reduced if historical results can substitute for new analyses. Collecting these data is only the initial step, however. Assessing their usefulness to the current project should always be performed before substantial effort is spent on incorporating historical results into a project database. If it is determined that the historical data are of questionable use for a specific project, then the determination of the most appropriate chemical analyses that will meet the project needs, including the level of effort necessary, will need to be assessed.

- **Spill data**—Evidence of a contaminant spill within or near the dredging area may be an important consideration in identifying locations for sampling.

- **Dredging history**—Knowledge of prior dredging may dramatically affect sampling plans. If the area is frequently dredged (every 1–2 years) or if the area is subject to frequent ship traffic, the sediments are likely to be relatively homogenous. Assuming that there is no major contaminant input, the sampling effort may be minimal. However, if there is information regarding possible contamination, a more extensive sampling effort may be indicated. New excavations of material unaffected by anthropogenic input may require less intensive sampling than maintenance dredging.

An acceptable sampling plan, including QA/QC requirements, should be in place before sampling begins. Regional guidance from governmental agencies (i.e., EPA and USACE) is required for developing these project-specific sampling plans. The sampling plan should be written so that a field sampling team unfamiliar with the site would be able to gather the necessary samples and field information.

Addressing quality assurance in the sampling plan includes designating field samples to be collected and used for assessing the quality of sampling and analysis, and ensuring that quality assurance is included in standard operating procedures for field measurements. The quality of the information obtained through the testing process is impacted by the following four factors:

- Collecting representative samples
- Collecting an appropriate number of samples
- Using appropriate sampling techniques
- Protecting or preserving the samples until they are tested.

Ideally, the importance of each of these four factors will be fully understood and appropriately implemented; in practice, however, this is not always the case. There may be occasions when time or other resource constraints will limit the amount of information that should or can be gathered. When this is the case, the relative importance of each of these factors has to be carefully considered in light of the specific study purposes.

An important component of any field sampling program is a preproject meeting with all concerned personnel. As with the drafting of the QA project plan, participation by several individuals may be necessary when developing the sampling plan. Personnel involved may include management, field personnel, laboratory personnel, data management/analysis personnel, and representatives of regulatory agencies, the permit applicant, and the dredging company. To assure sampling quality, at least one individual familiar with the study area should be included in the preproject meeting. The purposes of the meeting include:

- Defining the objectives of the sampling program
- Ensuring communication among participating groups
- Ensuring agreement on methods and contingency plans.

The more explicitly the objectives of a testing program can be stated (including QA objectives), the easier it will be to design an appropriate sampling plan. A complete sampling plan will result in a level of detail such that all sampling procedures and locations are clearly defined, sample volumes are clearly established, and all logistical concerns are fully addressed.

To ensure an adequate level of confidence in the data produced and in the comparability of the data to information collected by other sampling teams, the sampling plan should adhere to published sampling protocols and guidance. Descriptions of widely used sampling methods can be found in several EPA publications, many of which are cited in this section.

The sampling plan should include the following specific sections:

- Summary of dredging plan, including the dimensions of the dredging area, the dredging depths, side-slopes, and the volume of sediment for disposal (including overdredged material)
- Site background and existing database for the area, including identification of 1) relevant data, 2) need for additional data, and 3) areas of potential environmental concern within the confines of the dredging project
- Subdivision of dredging area into project segments, if appropriate, based on an assessment (review of historical data and past assessment work) of the level of environmental concern within the dredging area
- Sample location and sample collection frequency, including selection of methods and equipment for positioning vessels at established stations
- Sample designation system (i.e., a description of how each independently collected sample will be identified)
- Sample collection methods for all test media (e.g., sediment, water, or tissue)
- Procedures for sample handling (including container types and cleaning procedures), preservation, and storage, and (if applicable) field or shipboard analysis
- Logistical considerations and safety precautions.

The subsections that follow discuss each of these steps and provide general guidance for their conduct.

2.5.1 Review of Dredging Plan

A review of the plan for the dredging project provides a basis for determining the sampling strategy (see summary discussion in Section 2.3). The volume of material to be dredged and the method of dredging are two important factors that will help to determine the number of samples required. The number of samples required is generally a judgment that considers the cost, resolution,

and the risk of an incorrect decision regarding the volume of material to be dredged. Knowledge of the depth, volume, and physical characteristics of the material to be dredged will help to determine the kind of sampling equipment that is required. The boundaries of the dredging area have to be known (including the toe and the top of all side-slopes) to ensure that the number and location of samples are appropriate. Sampling should generally be to below the project depth plus any overdredging.

2.5.2 Site Background and Existing Database

As previously stated, reviewing the results of chemical analyses performed in past studies can help in selecting the appropriate contaminants of concern and in focusing plans for additional analyses. The level of data quality for historical data will affect the selection of station locations. Examples of four levels of data quality that can be assigned to historical results are summarized in Table 4. Labeling each set of results with a data quality level is also a simple way to summarize the relative quality of the data set for future use. This classification provides a useful summary of data quality when making conclusions and writing up the results of the project. The example classification in Table 4 considers the following factors when determining the suitability of historical results for a particular project:

- **The analytical methods used and their associated detection limits**—Analytical methods often improve over time. For example, as late as the 1970s, concentrations of many organic compounds in sediment samples were difficult to measure routinely, accurately, or sensitively. However, as better preparation methods and more sensitive analytical techniques have been developed, the ability to distinguish these compounds from other substances and the overall sensitivity of analyses have substantially improved. Methods are now available that afford detection limits that are well within the range of documented adverse biological effects.

- **QA/QC procedures and documentation**—The usefulness of data will depend on whether appropriate QA procedures have been used during analysis and if the data have been properly validated (see Section 2.9.1) and documented. Because more rigorous methods to analyze samples and document data quality have been required by environmental scientists over the past decade, only well-documented data that have been produced by laboratories using acceptable data quality controls should be considered to have no limitations. Historical data produced by even the best laboratories often may lack complete documentation, or the documentation may be difficult to obtain. However, historical data with incomplete documentation could still be used for projects with certain objectives (e.g., screening-level studies).

TABLE 4. LEVELS OF DATA QUALITY FOR HISTORICAL DATA

Level 1 **Data are acceptable for all project uses.**

The data are supported by appropriate documentation that confirms their comparability to data that will be generated in the current project.

Level 2 **Data are acceptable for most project uses.**

Appropriate documentation may not be available to confirm conclusions on data quality or to support legal defensibility. These data are supported by a summary of quality control information, and the environmental distribution of contamination suggested by these data is comparable to the distribution suggested by an independent analytical technique. The data are thus considered reliable and potentially comparable to data that will be produced in the project.

Level 3 **Data are acceptable for reconnaissance-level analyses.**

The data can be used to estimate the nature and extent of contamination. No supporting quality control information is available, but standard methods were used, and there is no reason to suspect a problem with the data based on 1) an inspection of the data, 2) their environmental distribution relative to data produced by an independent analytical technique, or 3) supporting technical reports. These data should be considered estimates and used only to provide an indication of the nature and possible extent of contamination.

Level 4 **Data are not acceptable for use in the current project.**

The data may have been acceptable for their original use. However, little or no supporting information is available to confirm the methods used, no quality control information is available, or there are documented reasons in technical reports that suggest the data may not be comparable to corresponding data to be collected in the current project.

2.5.3 Subdivision of Dredging Area

Sediment characteristics may vary substantially within the limits of the area to be dredged as a result of geographical and hydrological features. Many dredging projects can be subdivided into project segments (horizontal and/or vertical) that can be treated as separate management units. A project segment is an area expected to have relatively consistent characteristics that differ substantially from the characteristics of adjacent segments. Project segments may be sampled with various intensities as warranted by the study objectives and testing results.

Any established sampling program should be sufficiently flexible to allow changes based on field observations. However, any deviations from established procedures should be documented, along with the rationale for such deviations. An alteration checklist form is generally appropriate to implement required changes. An example of such a checklist is provided in Appendix A.

2.5.4 Sample Location and Collection Frequency

The method of dredging, the volume of sediment to be removed, the areal extent of the dredging project, and the horizontal and vertical heterogeneity of the sediment are key to determining station locations and the number of samples to be collected for the total dredging project. When appropriate to testing objectives, samples may be composited prior to analysis (with attention to the discussion in Section 2.5.4.8). The appropriate number of samples and the proper use of compositing should be determined for each operation on a case-by-case basis.

Using pertinent available information to determine station locations within the dredging area is both cost effective and technically efficient. If a review of historical data (see Section 2.5.2) identifies possible sources of contamination, skewing the sampling effort toward those areas may be justified to thoroughly characterize those areas, but can lead to an incomplete assessment of contamination in the whole study area. In areas of unequally distributed contamination, the total sampling effort should be increased to ensure representative, but not necessarily equal, sampling of the entire site. The following factors should be among those considered when selecting sampling stations and patterns: objectives of the testing program, bathymetry, area of the dredging project, accessibility, flows (currents and tides), mixing (hydrology), sediment heterogeneity, contaminant source locations, land use activities, available personnel and facilities, and other physical characteristics of the sampling site. A discussion of locating appropriate stations, sample collection, and sample handling procedures is provided in the following sections.

2.5.4.1 Station Locations

Station locations within the dredging area should include locations downstream from major point sources and in quiescent areas, such as turning basins, side channels, and inside channel bends, where fine-grained sediments and associated contaminants are most likely to settle. Information that should help to define the representativeness of stations within a dredging area includes:

- Clearly defined distribution of sediments to be dredged (i.e., project depth, overdredged depth, and side slopes)
- Clearly defined area to be sampled
- Correctly distributed sampling locations within each dredging area.

If sample variability is suspected within the dredging area, then multiple samples should be collected. When sediment variability is unknown, it may be necessary to conduct a preliminary survey of the dredging area to better define the final sampling program.

2.5.4.2 Sample Replication

Within a station, samples may be collected for replicate testing. Sediment testing is conducted on replicate samples, for which laboratory replicates (subsamples of a composite sample of the replicates) are generally recommended as opposed to field replicates (separate samples for each replicate). The former involves pseudo-replication but is more appropriate for dredged material evaluations where sediments will be homogenized by the dredging and discharge process. The latter involves true replication but is more appropriate for field investigations of the extent and degree of variability of sediment toxicity.

2.5.4.3 Depth Considerations

Sediment composition can vary vertically as well as horizontally. Samples should be collected over the entire dredging depth (including over-dredging), unless the sediments are known to be vertically homogenous or there are adequate data to demonstrate that contamination does not extend throughout the depth to be excavated. Separate analyses of defined sediment horizons or layers may be useful to determine the vertical distribution of contamination.

2.5.4.4 *Sampling Bias*

Ideally, the composition of an area and the composition of the samples obtained from that area will be the same. However, in practice, there often are differences due to bias in the sampling program, including disproportionate intensity of sampling in different parts of the dredging area and equipment limitations.

In some cases, to minimize bias, it may be useful to develop a sampling grid. The horizontal dimensions may be subdivided into grid cells of equal size, which are numbered sequentially. Cells are then selected for sampling either randomly or in a stratified random manner. It can be important to collect more than the minimum number of samples required, especially in areas suspected of having high or highly variable contamination. In some cases extra samples may be archived (for long time periods in the case of physical characterization or chemical analyses and for short time periods in the case of biological tests) should reexamination of particular stations be warranted.

In other cases, a sampling grid may not be desirable. This is particularly the case where dredging sites are not continuous open areas, but are rather a series of separate humps, bumps, reaches, and pockets with varying depths and surface areas. In these latter cases, sample distribution is commonly biased with intent.

2.5.4.5 *Level of Effort*

In some cases, it may be advisable to consider varying the level of sampling effort. Dredging areas suspected or known to be contaminated may be targeted for an increased level of effort so that the boundaries and characteristics of the contamination can be identified. A weighting approach can be applied whereby specific areas are ranked in increasing order of concern, and level of concern can then be used as a factor when determining the number of samples within each area.

2.5.4.6 *Number of Samples*

In general, the number of samples that should be collected within each dredging area is inversely proportional to the amount of known information, and is proportional to the level of confidence that is desired in the results and the suspected level of contamination. No specific guidance can be provided, but the following factors should be considered:

- The greater the number of samples collected, the better the areal and vertical definition

- Single measurements are inadequate to describe variability
- The means of several measurements at each station within a dredging area are generally less variable than individual measurements at each station.

2.5.4.7 Time and Funding Constraints

In all cases, the ultimate objective is to obtain sufficient information to evaluate the environmental impact of a dredged material disposal operation. The realities of time and funding constraints have to be recognized, although such do not justify inadequate environmental evaluation. Possible responses to cost constraints have been discussed by Higgins (1988). If the original sampling design does not seem to fit time or funding constraints, several options are available, all of which increase the risk of an incorrect decision. For example, the number of segments into which the project is divided can be reduced, but the total number of samples remains the same. This option results in fewer segments and maintains the power of station-to-station comparisons. This may, however, provide a poor assessment of spatial variability because of reduced stratification. Another example would be to maintain (or even increase) the number of stations sampled, and composite multiple samples from within a segment. This option results in a lower number of analyses being performed per segment, but may provide a poor assessment of spatial variability within each segment.

2.5.4.8 Sample Compositing

The objective of obtaining an accurate representation and definition of the dredging area has to be satisfied when compositing samples. Compositing provides a way to control cost while analyzing sediments from a large number of stations. Compositing results in a less detailed description of the variability within the area sampled than would individual analysis at each station. However if, for example, five analyses can be performed to characterize a project segment, the increased coverage afforded by collecting 15 individual samples and combining sets of three into five composite samples for analysis may justify the increased time and cost of collecting the extra 10 samples. Compositing can also provide the large sample volumes required for some biological tests. Composite samples represent the "average" of the characteristics of the individual samples making up the composite and are generally appropriate for logistical and other reasons; however, they are not recommended where they could serve to "dilute" a highly toxic but localized sediment "hot spot." Further, composite samples are not recommended for stations with very different grain size characteristics.

2.5.4.9 *Sample Definition*

When a sediment sample is collected, a decision has to be made as to whether the entire sediment volume is to be considered as the sample or whether the sediment volume represents separate samples. For instance, based on observed stratification, the top 1 m of a core might be considered to be a separate sample from the remainder of the core. After the sediment to be considered as a sample is identified, it should be thoroughly homogenized. Samples may be split before compositing, with a portion of the original sediment archived for possible later analysis, and the remainder combined with parts of other samples. These are then thoroughly homogenized (using clean instruments until color and textural homogeneity are achieved), producing the composite sample.

2.5.5 *Sample Designation System*

Information on the procedures used to designate the sampling location and type of sample collected should be clearly stated in the field sampling plan. The sampling stations should be named according to the site and the type of station. Each sample should be assigned an identifier that describes the station, type of sample, and field replicate. An example sample designation format is as follows:

- The first two characters of the station name could identify the site (e.g., BH = Boston Harbor).
- The third character of the station name could identify the type of station (e.g., S = site station, P = perimeter station, or R = reference station).
- The fourth and fifth characters of the station name could consist of a sequential number (e.g., 01, 02, or 03) that would be assigned to distinguish between different stations of the same type.
- The sixth character of the station name could describe the type of sample (e.g., C = sediment for chemistry and bioassay analyses, B = bioaccumulation, or I = benthic infauna).
- The resulting sample identifier would be: BHS01C.

When field replicates are collected (i.e., for benthic samples), the replicate number should be appended to the sample identifier. All field replicates from the same station should have the same sample identifier. The sample identifier and replicate number should be linked by a dash to form a single identifier for use on sample labels. The sample date should also be recorded on the sample label.

2.5.6 Station Positioning

The type of positioning system used during sample collection and detailed procedures for station positioning should be clearly stated in the sampling plan. No single positioning method will be appropriate for all sampling scenarios. U.S. EPA (1987b), PSEP(1990b), and USACE (1990) provide useful information on positioning systems and procedures. Guidance in these publications may be followed on all points that do not conflict with this document.

2.5.6.1 Selection of Station Positioning System

Available systems should be evaluated based on positioning requirements and project-specific constraints to select the most appropriate station positioning method for the project. Specific design and location factors that may affect station positioning include physical conditions (e.g., weather and currents) and topography of the study site, proposed equipment and analyses, minimum station separation, station reoccupation, and program-imposed constraints. U.S. EPA's (1993b) locational data policy implementation guidance calls for positioning accuracy within 25 m.

There are many methods available for navigating and positioning sampling vessels. These methods range from simple extensions of well-established onshore survey techniques using theodolites to highly sophisticated electronic positioning systems. A general discussion of a few of the station positioning methods available for dredged material evaluations is provided in the following sections. U.S. EPA (1987b), PSEP (1990b), USACE (1990), and current literature from the manufacturers of station positioning systems should be thoroughly reviewed during the selection process to choose the most appropriate project-specific positioning system.

Optical Positioning Techniques

Optical positioning requires visual sighting to determine alignment on two or more ranges, or the distances and angles between the vessel and shore targets.

Intersecting ranges can be used when a number of established landmarks permit easy selection of multiple ranges that intersect at the desired sampling point, and accuracy is not critical. One of the more traditional optical positioning systems is the theodolite system. Position of the sampling vessel can be established using theodolites by two onshore observers who simultaneously measure the angle between a reference object or shore traverse and the vessel. Using a theodolite with an accuracy of ±15 seconds for a single angle measurement at an intercept angle of approximately 45° and a

range of 5 km, could potentially yield a positioning error of < ±1 m (Ingham 1975). Although the accuracy of this method is good under optimal conditions, its use in open waters has several disadvantages such as limited line-of-sight, limits on intersection of angles, requirement of two manned shore stations, simultaneous measurements, and target movement and path interferences (e.g., fog, heavy rain, or heat waves).

Electronic Positioning Techniques

Electronic positioning systems use the transmission of electromagnetic waves from two or more stations and a vessel transmitter to define a vessel's location. Under routine sampling conditions, which may disfavor optical positioning, and at their respective maximum ranges, electronic positioning methods have greater accuracy than optical positioning methods (U.S. EPA 1987b).

LORAN-C is one type of electronic positioning system. Based on the signal properties of received transmissions from land-based transmitters, the LORAN-C receiver can be used to locate an approximate position, with a repeatable accuracy that varies from 15 to 90 m (U.S. EPA 1987b), depending on the weather and the geometry of the receiver within the LORAN-C station network. Although the LORAN-C system is not limited by visibility or range restrictions and does not require additional personnel to monitor onshore stations (as the theodolite system does), the LORAN-C system does experience interferences in some geographic areas and is more appropriately used to reposition on a previously sampled station.

Microwave positioning systems are typically effective between 25 and 100 km offshore, depending on antenna heights and power outputs, and have accuracies of 1-3 m. Microwave systems consist of two or more slave shore stations positioned over known locations and a master receiver on the vessel. By accurately measuring the travel time of the microwaves between the two known shore points and the vessel receiver, the position of the vessel can be accurately determined. The shore stations, typically tripod-mounted antennas powered by 12-volt batteries, are very susceptible to vandalism.

The global positioning system (GPS) is another electronic system that can determine station positions by receiving digital codes from three or more satellite systems, computing time and distance, and then calculating an earth-based position. Two levels of positioning accuracy are achievable with the GPS system. The positional accuracy of standard GPS is approximately 50–100 m (U.S. EPA 1987b). The accuracy can be improved to between 0.5–5 m by differential GPS (U.S. EPA 1987b). In differential GPS, two receivers are used. The master receiver is placed on a known location. It's location is computed based on satellite data, and a correction is applied to account for the errors in

position from the satellites. This correction is then sent via radio link or satellite to vessel-mounted receivers.

Hybrid Positioning Techniques

A number of hybrid positioning systems combine positional data from various sources to obtain fixes. Such systems usually involve the intersection of a visual line-of-position with an electronic line-of-position. Of particular interest to coastal monitoring programs are dynamic positioning systems that require only a single shore station and that use the simultaneous measurement of angle from a known direction and range to the survey vessel. These range-azimuth systems are characterized by their operating medium (optional, microwave, laser) and/or procedure (i.e. manual or automatic tracking).

2.5.6.2 Physical Conditions at the Study Site

The ability of a positioning method to achieve its highest projected accuracy depends, in part, on site-specific conditions. Weather, currents and other physical factors may reduce the achievable accuracy of a positioning method. For example, the relative drift of the sampling equipment away from the boat under strong currents or winds can increase with depth. Resulting positioning errors in sample location (as opposed to boat location) may exceed acceptable limits for the study if effects of site location on positioning accuracy are not considered during design of the sampling program.

2.5.6.3 Quality Assurance Considerations

Once the positioning method has been selected for the specific dredged material evaluation, the proper setup, calibration, and operational procedures must be followed to achieve the intended accuracy. At least one member of the field crew should be familiar with the selected positioning method.

Recordkeeping requirements should be established to ensure that station locations are accurately occupied and that adequate documentation is available. Adequate information to ensure consistent positioning and to allow reoccupation of stations for replicate sample collection or time-series monitoring should be kept in a field logbook. Entries should be initialed by the person entering the data. Required entries into the field logbook include the following:

- **Initial Survey Description**—The positioning method and equipment used, all changes or modifications to standard methods, names of persons who set up and operate the station positioning equipment, location of on-board equipment and the reference point (e.g., antennae, sighting position), the type of map used for

positioning and its identification number or scale should be recorded in the field logbook. In addition, a complete copy of the survey notes (if appropriate) should be included in the field logbook.

- **Day Log Entries**—The same information that is included in the initial survey description is also recorded on a daily basis in the day log. In addition, all problems or irregularities, any weather or physical conditions that may affect achievable accuracy, and all calibration data should be recorded in the day log.
- **Station Log Entries**—Each station location should be recorded in the coordinates or readings of the method used for positioning in sufficient detail to allow reoccupation of the station in the future. The positioning information should be recorded at the time of sample collection (versus time of equipment deployment) and for every reoccupation of that station, even during consecutive replicate sampling. In addition, supplemental positioning information that would define the station location or help subsequent relocation (e.g., anchored, tied to northwest corner of pier, buoy) should be recorded. If photographs are to be used for *a posteriori* plotting of stations, the roll and frame numbers should be recorded. Depth, time (tidal height) ship heading, and wire angle estimation should also be recorded for each occupation of a station.

Sampling reports should include the type of positioning method used during data collection. Any specific problems (e.g., wind, currents, waves, visibility, electronic interferences) that resulted in positioning problems and those stations affected should be identified in the sampling report. Estimates of the accuracy achieved for station positioning should be included. Station locations should be reported in appropriate units (e.g., latitude and longitude to the nearest second). Coordinates do not need to be reported for each replicate collected; a single set of coordinates for the station is sufficient. Depth corrected to mean lower low water should also be supplied for each station.

2.5.7 Sample Collection Methods

Detailed procedures for performing all sampling and equipment decontamination should be clearly stated in the sampling plan and can be included as standard operating procedures (see Appendix D). Sample collection requires an experienced crew, an adequate vessel equipped with navigational and supporting equipment appropriate to the site and the study, and noncontaminating sampling apparatus capable of obtaining relatively undisturbed and representative samples. To assure sampling quality, at least one individual familiar with the study area should be present during the

sampling activities. Sampling effort for a proposed dredging project is primarily oriented toward collection of sediment samples for physical and chemical characterization and for biological tests. Collection of water samples is also required to evaluate potential water column impact. Collection of organisms near the disposal site might be necessary if there is a need to characterize indigenous populations or to assess concentrations of contaminants in tissues. Organisms for use in toxicity and bioaccumulation tests may also be field-collected.

In general, a hierarchy for sample collection should be established to prevent contamination from the previous sample, especially when using the same sampling apparatus to collect samples for different analyses. Where possible, the known or expected least contaminated stations should be sampled first. At a station where water and sediment are to be collected, water samples should be collected prior to sediment samples. The vessel should ideally be positioned downwind or downcurrent of the sampling device. When lowering and retrieving sampling devices, care should be taken to avoid visible surface slicks and the vessel's exhaust. The deck and sample handling area should be kept clean to help reduce the possibility of contamination.

2.5.7.1 Sediment Sample Collection

Mudroch and MacKnight (1991) provide useful reference information for sediment sampling techniques. Higgins and Lee (1987) provide a perspective on sediment collection as commonly practiced by USACE. ASTM (1991b) and Burton (1991) provide guidelines for collecting sediments for toxicological testing. Guidance provided in these publications may be followed on all points that do not conflict with this document.

Care should be taken to avoid contamination of sediment samples during collection and handling. A detailed procedure for handling sampling equipment and sample containers should be clearly stated in the sampling plan associated with a specific project; this may be accomplished by using standard operating procedures. For example, samples designated for trace metal analysis should not come into contact with metal surfaces (except stainless steel, unless specifically prohibited for a project), and samples designated for organic analysis should not come into contact with plastic surfaces.

A coring device is recommended whenever sampling to depth is required. The choice of corer design depends on factors such as the objectives of the sampling program, sediment volumes required for testing, sediment characteristics, water depth, sediment depth, and currents or tides. A gravity corer may be limited to cores of 1–2 m in depth, depending on sediment grain size, degree of sediment compaction, and velocity of the drop. For penetration greater than 2 m, a vibratory corer or a piston corer is generally preferable. These types of coring devices are generally limited to soft, unconsolidated sediments. A split-spoon core may be used for more compacted sediment. The length of core that can be collected is usually limited to 10 core diameters in sand substrate and 20 core diameters in clay substrate. Longer cores can

be obtained, but substantial sample disturbance results from internal friction between the sample and the core liner.

Gravity corers can cause compaction of the vertical structure of sediment samples, if they freefall into the sediment. Therefore, if the vertical stratification in a core sample is of interest, a piston corer or vibra corer should be used. The piston corer uses both gravity and hydrostatic pressure. As the cutting edge penetrates the sediments, an internal piston remains at the level of the sediment/water interface, preventing sediment compression and overcoming internal friction. The vibra corer is a more complex piece of equipment but is capable of obtaining 3- to 7-m cores in a wide range of sediment types by vibrating a large diameter core barrel through the sediment column with little compaction. If the samples will not be sectioned prior to analysis, compaction is not a problem, and noncontaminating gravity (freefall) corers may be the simplest alternative.

Corers are the samplers of preference in most cases because of the variation in contamination with depth that can occur in sediment deposits. Substantial variation with depth is less likely in shallow channel areas without major direct contaminant inputs that have frequent ship traffic and from which sediments are dredged at short intervals. Generally, in these situations, bottom sediments are frequently resuspended and mixed by ship scour and turbulence, effectively preventing stratification. In such cases, surface grab samples can be representative of the mixed sediment column, and corers should be necessary only if excavation of infrequently disturbed sediments below the mixed layer is planned. Grab samplers are also appropriate for collecting surficial samples of reference or control sediments.

Grab samplers and gravity corers can either be Teflon®-coated or be made of stainless steel to prevent potential contamination of trace metal samples. The sampling device should at least be rinsed with clean water between samples. More thorough cleaning will be required for certain analyses; for instance, analyses performed for chlorinated dioxins require that all equipment and sample containers be scrupulously cleaned with pesticide-grade solvents or better because of the low detection limits required for these compounds. It is recommended that a detailed standard operating procedure specifying all decontamination procedures be included in the project sampling plan.

2.5.7.2 Water Sample Collection

If water samples are necessary, they should be collected with either a noncontaminating pump or a discrete water sampler. When sampling with a pump, the potential for contamination can be minimized by using a peristaltic or a magnetically coupled impeller-design pump. These kinds of pumps provide barriers between the sample and the surfaces of the pump (e.g., motor or fan)

that would cause contamination. The system should be flushed with the equivalent of 10 times the volume of the collection tubing. Also, any components within several meters of the sample intake should be noncontaminating (i.e., sheathed in polypropylene or epoxy-coated or made of Teflon®). Potential sample contamination must be avoided, including vessel emissions and other sampling apparatus.

A discrete water sampler should be of the close/open/close type so that only the target water sample comes into contact with internal sampler surfaces. Water samplers should be made of stainless steel or acrylic plastic. Seals should be Teflon®-coated whenever possible. Water sampling devices should be acid-rinsed (1:1 nitric acid) prior to use for collection of trace-metal samples, and solvent-rinsed (assuming the sampler material is compatible) prior to collection of samples for organic analyses.

2.5.7.3 Organism Collection

Collection methods for benthic organisms may be species-specific and can include, but are not limited to, bottom trawling, grabs, or cores. If organisms are to be maintained alive, they should be transferred immediately to containers with clean, well-oxygenated water, and sediment, as appropriate. Care must be taken to prevent organisms from coming into contact with natural predators and potentially contaminated areas or fuels, oils, natural rubber, trace metals, or other contaminants (U.S. EPA 1990a, 1992a).

2.5.8 Sample Handling, Preservation, and Storage

Detailed procedures for sampling handling, preservation, and storage should be part of the project-specific protocols and standard operating procedures specified for each sampling operation and included in the sampling plan. Samples are subject to chemical, biological, and physical changes as soon as they are collected. Sample handling, preservation, and storage techniques have to be designed to minimize any changes in composition of the sample by retarding chemical and/or biological activity and by avoiding contamination. Collection methods, volume requirements, container specifications, preservation techniques, storage conditions, and holding times (from the time of sample collection) for sediment, water, and tissue samples are discussed below and summarized in Table 5. Exceedance of the holding times presented in Table 5 would not necessarily result in qualification of the data during data validation. However, technical reasons justifying acceptance of data that exceed the holding time should be provided on a compound class basis.

TABLE 5. SUMMARY OF RECOMMENDED PROCEDURES FOR SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Analyses	Collection Method[a]	Sample Volume[b]	Container[c]	Preservation Technique	Storage Conditions	Holding Times[d]
Sediment						
Chemical/Physical Analyses						
Metals	Grab/corer	100 g	Precleaned polyethylene jar[e]	Dry ice[e] or freezer storage for extended storages; otherwise refrigerate	≤ 4°C	Hg - 28 days Others - 6 months[f]
Organic compounds (e.g., PCBs, pesticides, polycyclic aromatic hydrocarbons)	Grab/corer	250 g	Solvent-rinsed glass jar with Teflon® lid[e]	Dry ice[e] or freezer storage for extended storage; otherwise refrigerate	≤ 4°C[e]/dark[f]	14 days[g]
Particle size	Grab/corer	100 g	Whirl-pac bag[e]	Refrigerate	< 4°C	Undetermined
Total organic carbon	Grab/corer	50 g	Heat treated glass vial with Teflon®-lined lid[e]	Dry ice[e] or freezer storage for extended storages; otherwise refrigerate	≤ 4°C[e]	14 days
Total solids/specific gravity	Grab/corer	50 g	Whirl-pac bag	Refrigerate	< 4°C	Undetermined
Miscellaneous	Grab/corer	≥ 50 g	Whirl-pac bag	Refrigerate	< 4°C	Undetermined
Sediment from which elutriate is prepared	Grab/corer	Depends on tests being performed	Glass with Teflon®-lined lid	Completely fill and refrigerate	4°C/dark/airtight	14 days
Biological Tests						
Dredged material	Grab/corer	12–15 L per sample	Plastic bag or container[h]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[i]
Reference sediment	Grab/corer	45–50 L per test	Plastic bag or container[h]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[i]
Control sediment	Grab/corer	21–25 L per test	Plastic bag or container[h]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[i]

TABLE 5. (cont.)

Analyses	Collection Method[a]	Sample Volume[b]	Container[c]	Preservation Technique	Storage Conditions	Holding Times[d]
Water and Elutriate						
Chemical/Physical Analyses						
Particulate analysis	Discrete sampler or pump	500–2,000 mL	Plastic or glass	Lugols solution and refrigerate	4°C	Undetermined
Metals	Discrete sampler or pump	1 L	Acid-rinsed polyethylene or glass jar[j]	pH < 2 with HNO_3; refrigerate[j]	4°C 2°C[j]	Hg - 14 days Others - 6 months[k]
Total Kjeldahl nitrogen	Discrete sampler or pump	100–200 mL	Plastic or glass[k]	H_2SO_4 to pH < 2; refrigerate	4°C[k]	24 h[k]
Chemical oxygen demand	Discrete sampler or pump	200 mL	Plastic or glass[k]	H_2SO_4 to pH < 2; refrigerate	4°C[k]	7 days[k]
Total organic carbon	Discrete sampler or pump	100 mL	Plastic or glass[k]	H_2SO_4 to pH < 2; refrigerate	4°C[k]	<48 hours[k]
Total inorganic carbon	Discrete sampler or pump	100 mL	Plastic or glass[k]	Airtight seal; refrigerate[k]	4°C[k]	6 months[k]
Phenolic compounds	Discrete sampler or pump	1 L	Glass[k]	0.1–1.0 g $CuSO_4$; H_2SO_4 to pH < 2; refrigerate	4°C[k]	24 hours[k]
Soluble reactive phosphates	Discrete sampler or pump	--	Plastic or glass[k]	Filter; refrigerate[k]	4°C[k]	24 hours[k]
Extractable organic compounds (e.g., semivolatile compounds)	Discrete sampler or pump	4 L	Amber glass bottle[j]	pH < 2, 6N HCl; airtight seal; refrigerate	4°C[j]	7 days for extraction; 40 days for sample extract analyses[j]
Volatile organic compounds	Discrete sampler or pump	80 mL	Glass vial[j]	pH < 2 with 1:1 HCL; refrigerate in airtight, completely filled container[j]	4°C[j]	14 days for sample analysis, if preserved[j]
Total phosphorus	Discrete sampler or pump	--	Plastic or glass[h]	H_2SO_4 to pH < 2; refrigerate	4°C[k]	7 days[k]

TABLE 5. (cont.)

Analyses	Collection Method[a]	Sample Volume[b]	Container[c]	Preservation Technique	Storage Conditions	Holding Times[d]
Total solids	Discrete sampler or pump	200 mL	Plastic or glass[k]	Refrigerate	4°C[k]	7 days[k]
Volatile solids	Discrete sampler or pump	200 mL	Plastic or glass[k]	Refrigerate	4°C[k]	7 days[k]
Sulfides	Discrete sampler or pump	--	Plastic or glass[k]	pH > 9 NaOH (ZnAc); refrigerate[k]	4°C[k]	24 hours[k]
Biological Tests						
Site water	Grab	Depends on tests being performed	Plastic carboy	Refrigerate	< 4°C	14 days
Dilution water	Grab or makeup	Depends on tests being performed	Plastic carboy	Refrigerate	< 4°C	14 days
Tissue						
Metals	Trawl/Teflon®-coated grab	5–10 g	Double Ziploc®[e]	Handle with non-metallic forceps; plastic gloves; dry ice[e]	≤ –20°C[e] or freezer storage	Hg - 28 days Others - 6 months[m]
PCBs and chlorinated pesticides	Trawl/Teflon®-coated grab	10–25 g	Hexane-rinsed double aluminum foil and double Ziploc®[e]	Handle with hexane-rinsed stainless steel forceps; dry ice[e]	≤ –20°C[e] or freezer storage	14 days[g]
Volatile organic compounds	Trawl/Teflon®-coated grab	10–25 g	Heat-cleaned aluminum foil and water-tight plastic bag[l]	Covered ice chest[f]	≤ –20°C[m] or freezer storage	14 days[m]
Semivolatile organic compounds	Trawl/Teflon®-coated grab	10–25 g	Hexane-rinsed double aluminum foil and double Ziploc®[e]	Handle with hexane-rinsed stainless steel forceps; dry ice[e]	≤ –20°C[e] or freezer storage	14 days[g]
Lipids	Trawl/Teflon®-coated grab	Part of organic analyses	Hexane-rinsed aluminum foil	Handle with hexane-rinsed stainless steel forceps; quick freeze	≤ –20°C or freezer storage	14 days[g]

Note: This table contains only a summary of collection, preservation, and storage procedures for samples. The cited references should be consulted for a more detailed description of these procedures.

TABLE 5. (cont.)

PCB - polychlorinated biphenyl

[a] Collection method should include appropriate liners.

[b] Amount of sample required by the laboratory to perform the analysis (wet weight or volume provided, as appropriate). Miscellaneous sample size for sediment should be increased if auxiliary analytes that cannot be included as part of the organic or metal analyses are added to the list. The amounts shown are not intended as firm values; more or less tissue may be required depending on the analytes, matrices, detection limits, and particular analytical laboratory.

[c] All containers should be certified as clean according to U.S. EPA (1990c).

[d] These holding times are for sediment, water, and tissue based on guidance that is sometimes administrative rather than technical in nature. There are no promulgated, scientifically based holding time criteria for sediments, tissues, or elutriates. References should be consulted if holding times for sample extracts are desired. Holding times are from the time of sample collection.

[e] NOAA (1989).

[f] Tetra Tech (1986a).

[g] Sample may be held for up to 1 year if ≤ –20°C.

[h] Polypropylene should be used if phthalate bioaccumulation is of concern.

[i] Two weeks is recommended; sediments must not be held for longer than 8 weeks prior to biological testing.

[j] U.S. EPA (1987a); 40 CFR Part 136, Table III.

[k] Plumb (1981).

[l] If samples are not preserved to pH < 2, then aromatic compounds must be analyzed within 7 days.

[m] Tetra Tech (1986b).

2.5.8.1 *Sample Handling*

Sufficient sample volume should be collected to:

- Perform the necessary analyses
- Partition the samples, either in the field or as soon as possible after sampling, for respective storage and analytical requirements (e.g., freezing for trace metal analysis or refrigeration for bioassays)
- Archive portions of the sample for possible later analysis.
- Provide sample for replicate or QA analyses, if specified.

Sample handling is project- and analysis-specific, as well as being based on what is practical and possible. Generally, samples to be analyzed for trace metals should not come into contact with metals, and samples to be analyzed for organic compounds should not come into contact with plastics. All sample containers should be scrupulously cleaned (acid-rinsed for analysis of metals, solvent-rinsed for analysis of organic compounds).

For analysis of volatile compounds, samples should completely fill the storage container, leaving no airspace. These samples should be refrigerated but never frozen or the containers will crack. Samples for other kinds of chemical analysis are sometimes frozen. Only wide-mouth ("squat") jars should be used for frozen samples; narrow-mouth jars are less resistant to cracking. If the sample is to be frozen, sufficient air space should be left to allow expansion to take place (i.e., the wide-mouth sample container should be filled to no more than the shoulder of the bottle [just below the neck of the bottle] and the container should be frozen at an angle). Container labels have to withstand soaking, drying, and freezing without becoming detached or illegible. The labeling system should be tested prior to use in the field.

Sediment samples for biological testing should have larger (possible predatory) animals removed from the sediment by screening or press sieving prior to testing. Other matter retained on the screen with the organisms, such as shell fragments, gravel, and debris, should be recorded and discarded. Prior to use in bioassays, individual test sediments should be thoroughly homogenized with clean instruments (until color and textural homogeneity is achieved).

2.5.8.2 *Sample Preservation*

Preservation steps should be taken immediately upon sediment collection. There is no universal preservation or storage technique, although storage in the dark at 4°C is generally used for all samples held for any length of time prior to processing, and for some samples after processing. A technique for one group

of analyses may interfere with other analyses. This problem can be overcome by collecting sufficient sample volume to use specific preservation or storage techniques for specific analytes or tests. Preservation, whether by refrigeration, freezing, or addition of chemicals, should be accomplished as soon as possible after collection, onboard the collecting vessel whenever possible. If final preservation techniques cannot be implemented in the field, the sample should be temporarily preserved in a manner that retains its integrity.

Onboard refrigeration is easily accomplished with coolers and ice; however, samples should be segregated from melting ice and cooling water. Sediment samples that are to be frozen on board may be stored in an onboard freezer or may simply be placed in a cooler with dry ice or blue ice. Sample containers to be frozen (wide-mouth jars; see Section 2.5.7.1) should not be filled completely because expansion of the sample could cause the container to break. Sediment samples for biological analysis should be preserved at 4°C, never frozen or dried. Additional guidance on sample preservation is given in Table 5.

2.5.8.3 Sample Storage

The elapsed time between sample collection and analysis should be as short as possible. Sample holding times for chemical evaluations are analysis-specific (Table 5). Sediments for bioassay (toxicity and/or bioaccumulation) testing should be tested as soon as possible, preferably within 2 weeks of collection. Sediment toxicity does change with time. Studies to date suggest that sediment storage time should never exceed 8 weeks (at 4°C, in the dark, excluding air) (Becker and Ginn 1990; Tatem et al. 1991) because toxicity may change with storage time. Sample storage conditions (e.g., temperature, location of samples) should be documented.

2.5.9 Logistical Considerations and Safety Precautions

A number of frustrations in sample collection and handling can be minimized by carefully thinking through the process and requirements before going to the field. Contingency plans are essential. Well-trained, qualified, and experienced field crews should be used. Backup equipment and sampling gear, and appropriate repair parts, are advisable. A surplus of sampling containers and field data sheets should be available. Sufficient ice and adequate ice chest capacity should be provided, and the necessity of replenishing ice before reaching the laboratory should be considered. A vessel with adequate deck space is safer and allows for more efficient work than an overcrowded vessel. Unforeseeable circumstances (e.g., weather delays) are to be expected during field sampling, and time to adequately accommodate the unforeseen has to be included in sampling schedules.

Appropriate safety and health precautions must be observed during field sampling and sample processing activities. The EPA *Standard Operating Safety Guides* (U.S. EPA 1984b) should be used as a guidance document to prepare a site-specific health and safety plan. The health and safety plan should be prepared as a separate document from the QA project plan. Requirements implementing the Occupational Safety and Health Act at 29 CFR §1910.120 (Federal Register, Vol. 54, No. 43) should be met for medical surveillance, personal protection, respirator fit testing (if applicable), and hazardous waste operations training (if applicable) by all personnel working in contaminated areas or working with contaminated media.

The procedures and practices established in the site-specific health and safety plan should be observed by all individuals participating in the field activities. Safety requirements should also be met by all observers present during field audits and inspections. The plan should include the following information:

- Site location and history
- Scope of work
- Site control
- Hazard assessment (chemical and physical hazards)
- Levels of protection and required safety equipment
- Field monitoring requirements
- Decontamination
- Training and medical monitoring requirements
- Emergency planning and emergency contacts.

2.6 SAMPLE CUSTODY

Recordkeeping procedures are described in detail in this section of the QA project plan, including specific procedures to document the physical possession and condition of samples during their transport and storage. This section also describes how excess or used samples will be disposed of at the end of the project.

2.6.1 Sample Custody and Documentation

Sample custody and documentation are vital components of all dredged material evaluations, particularly if any of the data may be used in a court of law. It is important to record all events associated with a sample so that the validity of the resulting data may be properly interpreted. Thorough

documentation provides a means to track samples from the field through the laboratory and prevent sample loss. The contents and location of all documents related to dredged sediment samples should be specified, and access to the samples should be controlled.

The possession of samples should be documented from sample collection through laboratory analysis. Recording basic information during sample handling is good scientific practice even if formal custody procedures are not required. Sample custody procedures, including examples of forms to be used, should be described in the QA project plan. Minimum requirements for documentation of sample handling and custody on simple projects should include the following information:

- Sample location, project name, and unique sample number
- Sample collection date (and time if more than one sample may be collected at a location in a day)
- Any special notations on sample characteristics or problems
- Initials of the person collecting the sample
- Date sample sent to the laboratory
- Conditions under which the samples were sent to the laboratory.

For large or sensitive projects that may result in enforcement actions or other litigation, a strict system for tracking sample custody should be used to assure that one individual has responsibility for a set of samples at all times. For these projects, only data that have clear documentation of custody can be accepted without qualification.

A strict system of sample custody implies the following conditions:

- The sample is possessed by an individual and secured so that no one can tamper with it
- The location and condition of the sample is known and documented at all times
- Access to the sample is restricted to authorized personnel only.

Where samples may be needed for potential litigation, chain-of-custody procedures should be followed. Chain-of-custody procedures are initiated during sample collection. Chain-of-custody forms are often used to document the transfer of a sample from collection to receipt by the laboratory (or between different facilities of one laboratory). Although not always required, these forms provide an easy means of recording information that may be useful weeks or months after sample collection. When these forms are used, they are provided

to field technicians at the beginning of a project. The completed forms accompany the samples to the laboratory and are signed by the relinquisher and receiver every time the samples change hands. After sample analysis, the original chain-of-custody form is returned by the laboratory. The form is filed and becomes part of the permanent project documentation. An example of a chain-of-custody form is provided in Appendix A. Additional custody requirements for field and laboratory operations should be described in the QA project plan, when appropriate.

When in doubt about the level of documentation required for sampling and analysis, a strict system of documentation using standard forms should be used. Excess documentation can be discarded; lack of adequate documentation in even simple projects sometimes creates the unfortunate impression that otherwise reasonable data are unusable or limited. Formal chain-of-custody procedures are outlined briefly in the statements of work for laboratories conducting analyses of organic and inorganic contaminants under EPA's Contract Laboratory Program (CLP) (U.S. EPA 1990d,e).

2.6.1.1 Field Operations

The potential for sample deterioration and/or contamination exists during sample collection, handling, preservation, and storage. Approved protocols and standard operating procedures should be followed to ensure all field sampling equipment is acceptably calibrated and to prevent deterioration or contamination. Experienced personnel should be responsible for maintaining the sample integrity from collection through analysis, and field operations should be overseen by the project manager. A complete record of all field procedures, an inventory log, and a tracking log should be maintained. A field tracking report (see example in Appendix A) should identify sample custody and conditions in the field prior to shipment.

Dates and times of collection, station locations, sampling methods, and sample handling, preservation, and storage procedures should be documented immediately, legibly, and indelibly so that they are easily traceable. Any circumstances potentially affecting sampling procedures should be documented. The data recorded should be thorough enough to allow station relocation and sample tracking. An example of a station location log is provided in Appendix A. Any field preparation of samples should also be described. In addition, any required calibration performed for field instruments should be documented in the field logbook. Samples should be identified with a previously prepared label (see example in Appendix A) containing at least the following information:

- Project title
- Sample identification number

- Location (station number) and depth
- Analysis or test to be performed
- Preservation and storage method
- Date and time of collection
- Special remarks if appropriate
- Initials of person collecting the sample
- Name of company performing the work.

2.6.1.2 Laboratory Operations

Documentation is necessary in the laboratory where chemical and biological analyses are performed. A strict system of sample custody for laboratory operations should include the following items:

- Appointment of a sample custodian, authorized to check the condition of and sign for incoming field samples, obtain documents of shipment, and verify sample custody records
- Separate custody procedures for sample handling, storage, and disbursement for analysis in the laboratory
- A sample custody log consisting of serially numbered, standard laboratory tracking report sheets.

A laboratory tracking report (Appendix A) should be prepared for each sample. The location of samples processed through chain-of-custody must be known at all times. Samples to be used in a court of law must be stored in a locked facility to prevent tampering or alteration.

A procedure should be established for the retention of all field and laboratory records and samples as various tasks or phases are completed. Replicates, subsamples of analyzed samples, or extra unanalyzed samples should be kept in a storage bank. These samples can be used to scrutinize anomalous results or for supplemental analyses, if additional information is needed. All samples should be properly stored and inventoried. The retention and archiving procedure should indicate the storage requirements, location, indexing codes, retention time, and security requirements for samples and data.

2.6.2 Storage and Disposal of Samples

In the statement of work, the laboratory should be instructed to retain all remaining sample material (under appropriate temperature and light conditions) at least until after the QA review has been completed. In addition, sample extracts or digestates should be appropriately stored until disposal is approved by the project manager. With proper notice, most laboratories are willing to provide storage for a reasonable time period (usually on the order of weeks) following analysis. However, because of limited space at the laboratory, the project manager may need to make arrangements for long-term storage at another facility.

Samples must be properly disposed when no longer needed. Ordinary sample-disposal methods are usually acceptable, and special precautions are seldom appropriate. Under Federal law [40 CFR 261.5(a)], where highly contaminated wastes are involved, if the waste generated is less than 100 Kg per month, the generator is conditionally exempt as a small-quantity generator and may accumulate up to 1,000 Kg of waste on the property without being subject to the requirements of Federal hazardous waste regulations. However, State and local regulations may require special handling and disposal of contaminated samples. When samples have to be shipped, 49 CFR 100-177 should be consulted for current Department of Transportation regulations on packing and shipping.

Over the last few years, there has been a growing awareness of the ecological and economic damage caused by introduced species. Because both east and west coast species are often used in bioaccumulation tests, there is a real potential of introducing bioaccumulation test species or associated fauna and flora (e.g., pathogens, algae used in transporting the worms). It is the responsibility of the persons conducting the bioaccumulation or toxicity tests to assure that no non-indigenous species are released. The general procedures to contain non-indigenous species are to collect and then poison all water, sediment, organisms and associated packing materials (e.g., algae, sediment) before disposal. Chlorine bleach can be used as the poison. A double containment system is used to keep any spillage from going down the drain. Guidance on procedures used in toxicity tests can be found in Appendix B of DeWitt et al. (1992a). Flow-through tests can generate large quantities of water, and researchers should plan on having sufficient storage facilities.

2.7 CALIBRATION PROCEDURES AND FREQUENCY

Procedures for minimizing bias and properly maintaining the precision of each piece of equipment to be used in the field or laboratory are detailed in this section of the QA project plan. Procedures are also described for obtaining, using, and storing chemical standards of known purity used to quantify

analytical results, and reference chemicals used as positive controls in toxicity tests. Instruments that require routine calibration include, for example, navigation devices, analytical balances, and water quality meters.

Calibration of analytical instruments is a high priority and is always required for any project requiring quantitative data (even if only estimated quantities are necessary). Calibration is essential because it is the means by which instrument responses are properly translated into chemical concentrations. Instrument calibration is performed before sample analysis begins and is continued during sample analysis at intervals specified in each analytical method to ensure that the data quality objectives established for a project are met.

Because there are several analytical techniques that can be used for the same target analyte, each of which may provide different guidance for performing instrument calibration, it is important to establish a minimum calibration procedure for any chemical analysis that will be performed. Uniform adherence to a minimum calibration procedure will also improve the comparability of data generated by multiple laboratories that may be used for a specific project or among projects. All requirements for performing instrument calibrations should be clearly stated in the QA project plan and the laboratory statement of work prepared for any project.

In addition to performing instrument calibrations, the acceptability of the calibrations performed should be evaluated. To provide control over the calibration process, specific guidelines should be specified. The basic elements of the calibration process include the calibration frequency, number of calibration standards and their concentrations, and the calibration acceptance criteria. A summary of these elements is provided below.
Examples of the differences in calibration procedures (specifically for the analysis of organic compounds) for different analytical methods are provided in Table 6.

2.7.1 Calibration Frequency

The general process of verifying that an instrument is functioning acceptably is to perform initial and continuing calibrations. Initial calibration should be performed prior to sample analysis to determine whether the response of the instrument is linear across a range of target analyte concentrations (i.e., the working linear range). In addition to establishing the initial calibration for an instrument, it is critical that the stability of the instrument response be verified during the course of ongoing sample analyses. The verification of instrument stability is assessed by analyzing continuing calibration standards at regular intervals during the period that sample analyses are performed. Although each analytical method provides guidance for the frequency at which continuing

TABLE 6. EXAMPLE CALIBRATION PROCEDURES

Calibration Criteria	SW-846 Methods for Organic Compounds[a]	EPA CLP Methods for Organic Compounds[b]
Number of standards for initial calibration	Minimum of five for all methods	Five for all GC/MS analyses Three for pesticides One for PCBs and multicomponent pesticides
Concentration of lowest initial calibration standard	All target analytes near, but above, the TDL	Contractually set (e.g., 10 µg/L for volatile organic compounds)
Concentrations for initial calibration to establish the instrument's working linear range	1. Bracket the expected concentration range of analytes expected in samples 2. Bracket the full instrument/detector linear range	Contractually set (e.g., 10, 50, 100, 150, and 200 µg/L for volatile organic compounds)
Concentration of continuing calibration standards	Not specified, except for GC/MS methods	Contractually set (e.g., 50 µg/L for all GC/MS analyses)
Frequency of calibrations	Repeat when acceptance criteria not met	Repeat when acceptance criteria not met
Acceptance criteria for initial calibration[c]	Calculate analyte RRFs or RFs, then RSD should be ≤ 30 percent for GC/MS methods and ≤ 20 percent for all other methods Alternative: generate a least squares linear regression (peak height/area vs. concentration) and use equation to calculate sample results	Calculate analyte RRFs or RFs, then RSD should be ≤ 30 percent for GC/MS methods and ≤ 20 percent for pesticides Alternative: none
Acceptance criteria for continuing calibration[c]	Calculate analyte RRFs or RFs, then difference to mean RRF or RF of initial calibration should be ≤ 25 percent for GC/MS methods and ≤ 15 percent for all other methods Alternative: none	Calculate analyte RRFs or RFs, then difference to mean RRF or RF of initial calibration should be ≤ 25 percent for GC/MS methods and ≤ 15 percent for pesticides Alternative: none

Note: CLP - Contract Laboratory Program
GC/MS - gas chromatography/mass spectrometry
PCB - polychlorinated biphenyl
RF - response factor (i.e., calibration factor)
RRF - relative response factor
RSD - relative standard deviation
TDL - target detection limit

TABLE 6. (cont.)

[a] U.S. EPA (1986a).

[b] U.S. EPA (1990b).

[c] The acceptance criteria for instrument calibration (i.e., initial and continuing calibration) may not be available for all organic compounds listed in Table 3 (e.g., resin acids and guaiacols). The determination of acceptable instrument calibration criteria for organic compounds not specifically stipulated in SW-846 or EPA CLP methods should be assessed using best professional judgment (e.g., ≤ 50 percent RSD).

calibration standards should be performed, it is recommended that at a minimum these standards be analyzed at the beginning of each analytical for table 6 sequence, after every tenth sample, and at the end of the analytical sequence for all organic and inorganic compound analyses performed. The concentration of the continuing calibration standard should be equivalent to the concentration of the midpoint established during initial calibration of the working linear range of the instrument.

2.7.2 Number of Calibration Standards

Specific instrument calibration procedures are provided in most analytical methods; however, a wide variation exists in the number of calibration standards specified for different analyses. To ensure that consistent and reliable data are generated, a minimum number of calibration standards should be required for all laboratories performing chemical analyses.

Typically, as the number of calibration standards increases, the reliability of the results increases for concentrations detected above the TDL. The specific standards that are selected for calibration can have a significant impact on the validity of the data generated. Calibration standards should be established with respect to the range of standards required, the TDLs selected, and the linear range of the target analytes desired. Specific requirements for establishing the number of calibration standards, including recommendations on the concentrations to use, will be different for organic and inorganic analyses; however, some general recommended guidelines are provided below.

The working linear range of an instrument should be established prior to performing sample analyses. A minimum of five calibration standards for the analysis of organic compounds and three calibration standards for the analysis of inorganic compounds should be used when establishing the working linear range for all target analytes of concern. Generally, the working linear range of an instrument for a specific analysis should bracket the expected concentrations of the target analyte in the samples to be analyzed. In some instances, however, it may not be known what analyte concentrations to expect. A 5-point initial calibration sequence is recommended to establish the working linear range for organic chemical analyses.

In addition to the number of standards analyzed, the difference between the concentration of the lowest standard and the TDL and the difference between each standard used to establish the initial calibration are critical. The selection of the lowest initial calibration standard concentration will provide more confidence in the documented bias of results reported as undetected at the TDL or any results reported at very low concentrations. The selection of this standard will also ensure that target analytes can be reliably detected above instrument background noise and potential matrix interferences. For the

dredged material program, this standard should be no lower than the TDL provided in Table 3.

The decision as to which specific concentrations (i.e., calibration range) should be used for a multipoint calibration requires careful consideration. While methods established by EPA CLP protocols provide stringent requirements for calibration analyses, these requirements are not clearly specified for other analytical methods (e.g., SW-846 methods) (see Table 6). A 5-point initial calibration sequence is recommended for all non-CLP methods. The concentrations of all standards should range from the lowest concentration meeting the requirements suggested above to the highest standard concentration equivalent to the upper linear range of the instrument/detector configuration. The concentrations of the remaining three standards should be evenly distributed between these concentrations. The calibration standards used to establish the working linear range should encompass a factor of 20 (i.e., 1 to 20, with the lowest concentration equal to 1 and the highest concentration equal to 20 times the concentration of the lowest concentration used).

2.7.3 Calibration Acceptance Criteria

Once the initial calibration has been performed, the acceptability of the calibration should be assessed to ensure that the bias of the data generated will be acceptable; this assessment should be performed by all laboratories prior to the analysis of any sample. In addition, the acceptability of all continuing calibrations should be assessed.

Although each analytical method provides guidance for determining the acceptability of instrument calibrations, there are multiple options available (e.g., least squares linear regression, percent relative standard deviations, and percent differences). A specific set of acceptance criteria should be determined prior to sample analysis, and these criteria should be contractually binding to avoid unnecessary qualification or rejection of the data generated. A summary of the most widely used calibration acceptance criteria currently in use for organic analyses is provided in Table 6. Calibration acceptance criteria should be used to assess the acceptability of the initial calibration sequence in terms of the relationship between the intercept of the calibration curve (i.e., the x-y intercept) and the predetermined TDLs and the overall reliability of the working linear range established.

The general criteria specified by SW-846 methods are typically more stringent for organic analyses than the EPA CLP requirements. Acceptance criteria, as summarized in Table 6, should be clearly defined before sample analyses are performed. All specific acceptance criteria for calibrations should be stated in the QA project plan and the laboratory statement of work.

2.8 ANALYTICAL PROCEDURES

The methods cited in this section may be used to meet general data quality objectives for dredged material evaluations. However, other methods may provide similar results, and the final choice of analytical procedures should be based on the needs of each evaluation. In all cases, proven, current methods should be used; EPA-approved methods, if available, are preferred. Sample analysis procedures are identified in this section by reference to established, standard methods. Any modifications to these procedures and any specialized, nonstandard procedures are also described in detail. When preparing a QA project plan, only modifications to standard operating procedures or details of non-standard procedures need to be described in this section of the plan.

Any dredged material from estuarine or marine areas contains salt, which can interfere with the results obtained from some analytical methods. Any methods proposed for the analysis of sediment and water from estuarine or marine environments should explicitly address steps taken to control salt interference.

The following sections provide guidance on the selection of physical and chemical analyses to aid in evaluating dredged material proposed for disposal, and on the methods used to analyze these parameters. Information on the chemicals on the EPA priority pollutant and hazardous substance lists is provided in Appendix E.

2.8.1 Physical Analysis of Sediment

Physical characteristics of the dredged material must be determined to help assess the impact of disposal on the benthic environment and the water column and to help determine the appropriate dredging methods. This is the first step in the overall process of sediment characterization, and also helps to identify appropriate control and reference sediments for biological tests. In addition, physical analyses can be helpful in evaluating the results of analyses and tests conducted later in the characterization process.

The general analyses may include grain size distribution, total solids content, and specific gravity. Grain size analysis defines the frequency distribution of the size ranges of the particles that make up the sediment (e.g., Plumb 1981; Folk 1980). The general size classes of gravel, sand, silt, and clay are the most useful in describing the size distribution of particles in dredged material samples. Use of the Unified Soil Classification System (USCS) for physical characterization is recommended for the purpose of consistency with USACE engineering evaluations (ASTM 1992).

Measurement of total solids is a gravimetric determination of the organic and inorganic material remaining in a sample after it has been dried at a specified

temperature. The total solids values generally are used to convert concentrations of contaminants from a wet-weight to a dry-weight basis.

The specific gravity of a sample is the ratio of the mass of a given volume of material to an equal volume of distilled water at the same temperature (Plumb 1981). The specific gravity of a dredged material sample helps to predict the behavior (i.e., dispersal and settling characteristics) of dredged material after disposal.

Other physical/engineering properties (e.g., Atterburg limits, settling properties) may be needed to evaluate the quality of any effluent discharged from confined disposal facilities. QA considerations for physical analysis of sediments are summarized in Section 2.10.3.

2.8.2 Chemical Analysis of Sediment

Chemical analysis provides information about the chemicals present in the dredged material that, if biologically available, could cause toxicity and/or be bioaccumulated. This information is valuable for exposure assessment and for deciding which of the contaminants present in the dredged material to measure in tissue samples. This section discusses the selection of target analytes and techniques for sediment analyses. QA considerations are summarized in Section 2.10.4.

2.8.2.1 Selection of Target Analytes

If the review of data from previous studies suggests that sediment contaminants may be present (see Section 2.5.2), but fails to produce sufficient information to develop a definitive list of potential contaminants, a list of target analytes should be compiled. Target analytes should be selected from, but not necessarily limited to, those listed in Table 3. The target analyte list should also include other contaminants that historical information or commercial and/or agricultural applications suggest could be present at a specific dredging site (e.g., tributyltin near shipyards, berthing areas, and marinas where these compounds have been applied). Analysis of polycyclic aromatic hydrocarbons (PAHs) in dredged material should focus on those PAH compounds listed in Table 3.

All PCB analyses should be made using congener-specific methods. The sum of the concentrations of specific congeners is an appropriate measure of total PCBs (NOAA, 1989). Congener-specific analyses also provide data that can be used for specialized risk assessments that reflect the widely varying toxicity of different PCB congeners.

Sediments should be analyzed for TOC. This is particularly important if there are hydrophobic organic compounds on the target analyte list. The TOC content of sediment is a measure of the total amount of oxidizable organic material in a sample and also affects contaminant bioaccumulation by, and effects to, organisms (e.g., DeWitt et al. 1992b; Di Toro et al. 1991).

Sediments in which metals are suspected to be contaminants of concern may also be analyzed for acid volatile sulfide (AVS) (Di Toro et al. 1990; U.S. EPA 1991a). Although acceptable guidance on the interpretation of AVS measurements is not yet available, and AVS measurements are not generally required at this time, such measurements can provide information on the bioavailability of metals in anoxic sediments.

2.8.2.2 Selection of Analytical Techniques

Once the list of project-specific target analytes for sediments has been established, appropriate analytical methods should be determined (see Section 2.3). The analytical methods selected must be able to meet the TDLs established to meet the requirements of the intended uses of the data. Also, the methods selected will, to some degree, dictate the amount of sediment sample required for each analysis. Examples of methods that can be used to meet TDLs for dredged material evaluations are provided in Table 3. General sample sizes are provided in Table 5, and include possible requirements for more than one analysis for each group of analytes. The amount of sample used in an analysis affects the detection limits attainable by a particular method. The following overview summarizes various factors to be considered when selecting analytical methods for physical, inorganic, and organic analyses.

TOC analyses should be based on high-temperature combustion rather than on chemical oxidation, because some classes of organic compounds are not fully degraded by chemical/ultraviolet techniques. The volatile and nonvolatile organic components make up the TOC of a sample. Because inorganic carbon (e.g., carbonates and bicarbonates) can be a significant proportion of the total carbon in some sediment, the sample has to be treated with acid to remove the inorganic carbon prior to TOC analysis. The method of Plumb (1981) recommends the use of hydrochloric acid. An alternative choice might be sulfuric acid because it is nonvolatile, is used as the preservative, and does not add to the chloride burden of the sample. However, some functional groups (e.g., carboxylic acids) can be oxidized when inorganic carbonates are removed using both a non-oxidizing and an oxidizing acid. Whatever acid is used, it has to be demonstrated on sodium chloride blanks (for all marine samples) that there is no interference generated from the combined action of acid and salt in the sample. Acceptable methods for TOC analysis are provided in PSEP (1986) and U.S. EPA (1992b).

For many metals analyses in marine/estuarine areas, the concentration of salt may be much greater than the concentration of the analyte of interest, and can cause unacceptable interferences in certain analytical techniques. In such cases, the freshwater approach of acid digestion followed by inductively coupled plasma-atomic emission spectrometry (ICP) or graphite furnace atomic absorption spectrometry (GFAA) should be coupled with appropriate techniques for controlling this interference. For example, the mercury method in U.S. EPA (1986a; Method 7471) may be used for the analysis of mercury in sediment. Tributyltin may be analyzed by the methods of Rice et al. (1987) and NCASI (1986), and selenium and arsenic by the method of EPRI (1986). Total digestion of metals is not necessary for dredged material evaluations, although this technique is used for complete chemical characterizations in some national programs (e.g., NOAA Status and Trends). The standard aqua regia extraction yields consistent and reproducible results. The recommended method for analysis of semivolatile and volatile priority pollutants in sediments is described in Tetra Tech (1986a), and is a modified version of established EPA analytical methods designed to achieve lower and more reliable detection limits. Analysis for organic compounds should always use capillary-column gas chromatography (GC): gas chromatography/mass spectrometry (GC/MS) techniques for semivolatile and volatile priority pollutants, and dual column gas chromatography/electron-capture detection (GC/ECD) for pesticides and PCBs (NOAA 1989). Alternatively, GC/MS using selected ion monitoring can be used for PCB and pesticide analysis. These analytically sound techniques yield accurate data on the concentrations of chemicals in the sediment matrix. The analytical techniques for semivolatile organic compounds generally involve solvent extraction from the sediment matrix and subsequent analysis, after cleanup, using GC or GC/MS. Extensive cleanup is necessitated by the likelihood of 1) biological macromolecules, 2) sulfur from sediments with low or no oxygen, and 3) oil and/or grease in the sediment. The analysis of volatile organic compounds incorporates purge-and-trap techniques with analysis by either GC or GC/MS. If dioxin (i.e., 2,3,7,8-tetrachlorodibenzo-*p*-dioxin [TCDD]) analysis is being performed, the methods of Kuehl et al. (1987), Smith et al. (1984), U.S. EPA (1989b; Method 8290), or U.S. EPA (1990f; Method 1613) should be consulted. EPA Method 1613 is the recommended procedure for measuring the tetra- through octa- polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). This method has been developed for analysis of water, soil, sediment, sludge, and tissue. Table 7 shows the 17 compounds determined by Method 1613.

Techniques for analysis of chemical contaminants have some inherent limitations for sediment samples. Interferences encountered as part of the sediment matrix, particularly in samples from heavily contaminated areas, may limit the ability of a method to detect or quantify some analytes. The most selective methods using GC/MS techniques are recommended for all nonchlorinated organic compounds because such analysis can often avoid

TABLE 7. PCDD and PCDF Compounds Determined by Method 1613

Native Compound[1]
2,3,7,8-TCDF
2,3,7,8-TCDD
1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF
1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF
2,3,4,6,7,8-HxCDF
1,2,3,4,7,8-HxCDD
1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD
1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDF
1,2,3,4,6,7,8-HpCDD
1,2,3,4,7,8,9-HpCDF
OCDD
OCDF

[1] Polychlorinated Dioxins and Furans

TCDD = Tetrachlorodibenzp-p-dioxin
TCDF = Tetrachlorodibenzofuran
PeCDD = Pentachlorodibenzo-p-dioxin
PeCDF = Pentachlorodibenzofuran
HxCDD = Hexachlorodibenzo-p-dioxin
HxCDF = Hexachlorodibenzofuran
HpCDD = Heptachlorodibenzo-p-dioxin
HpCDF = Heptachlorodibenzofuran
OCDD = Octachlorodibenzo-p-dioxin
OCDF = Octachlorodibenzofuran

problems due to matrix interferences. GC/ECD methods are recommended by the EPA as the primary analytical tool for all PCB and pesticide analyses because GC/ECD analysis (e.g., NOAA 1989) will result in lower detection limits. The analysis and identification of PCBs by GC/ECD methods are based upon relative retention times and peak shapes. Matrix interferences may result in the reporting of false negatives, although the congener-specific PCB analysis reduces this concern relative to use of the historical Aroclor®-matching procedure.

For dredged material evaluations, the concentration of total PCBs should be determined by summing the concentrations of specific individual PCB congeners identified in the sample (see Table 8). The minimum number of PCB congeners that should be analyzed are listed in the first column of Table 7 (i.e., "summation" column) (NOAA 1989). This summation is considered the most accurate representation of the PCB concentration in samples. Additional PCB congeners are also listed in Table 8. McFarland and Clarke (1989) recommend these PCB congeners for analysis based on environmental abundance, persistence, and biological importance. Sample preparation for PCB congener analysis should follow the techniques described in Tetra Tech (1986a) or U.S. EPA (1986a), but with instrumental analysis and quantification using standard capillary GC columns on individual PCB isomers according to the methods reported by NOAA (1989) (see also Dunn et al. 1984; Schwartz et al. 1984; Mullin et al. 1984; Stalling et al. 1987).

Although the methods mentioned above are adequate for detecting and quantifying concentrations of those PCB congeners comprising the majority of total PCBs in environmental samples, they are not appropriate for separating and quantifying PCB congeners which may coelute with other congeners and/or may be present at relatively small concentrations in the total PCB mixture. Included in this latter group of compounds, for example, are PCBs 126 and 169, two of the more toxic nonortho-substituted PCB congeners (Table 8). In order to separate these (and other toxic nonortho-substituted congeners), it is necessary to initially utilize an enrichment step with an activated carbon column (Smith 1981). Various types of carbon columns have been used, ranging from simple gravity columns (e.g., in a Pasteur pipette) to more elaborate (and efficient) columns using high-pressure liquid chromatography (HPLC) systems (see Schwartz et al. 1993). The preferred method of separation and quantitation of the enriched PCB mixture has been via high resolution GC/MS with isotope dilution (Kuehl et al. 1991; Ankley et al. 1993; Schwartz et al. 1993). However, recent studies have shown that if the carbon enrichment is done via HPLC, the nonortho-substituted PCB congeners of concern also may be quantifiable via more widely available GC/ECD systems (Schwartz et al. 1993).

TABLE 8. POLYCHLORINATED BIPHENYL CONGENERS RECOMMENDED FOR QUANTITATION AS POTENTIAL CONTAMINANTS OF CONCERN

PCB Congener[a]	Congener Number[b]		
	Summation[c]	Highest Priority[d]	Second Priority[e]
2,4'-Dichlorobiphenyl	8		
2,2',5-Trichlorobiphenyl	18		18
2,4,4'-Trichlorobiphenyl	28		
3,4,4'-Trichlorobiphenyl			37
2,2',3,5'-Tetrachlorobiphenyl	44		44
2,2',4,5'-Tetrachlorobiphenyl			99
2,2',5,5'-Tetrachlorobiphenyl	52		52
2,3',4,4'-Tetrachlorobiphenyl	66		
2,3',4',5-Tetrachlorobiphenyl			70
2,4,4',5-Tetrachlorobiphenyl			74
3,3',4,4'-Tetrachlorobiphenyl	77	77	
3,4,4',5-Tetrachlorobiphenyl			81
2,2',3,4,5'-Pentachlorobiphenyl		87	
2,2',3,4',5-Pentachlorobiphenyl		49	
2,2',4,5,5'-Pentachlorobiphenyl	101	101	
2,3,3',4,4'-Pentachlorobiphenyl	105	105	
2,3,4,4',5-Pentachlorobiphenyl			114
2,3',4,4',5-Pentachlorobiphenyl	118	118	
2,3',4,4',6-Pentachlorobiphenyl			119
2',3,4,4',5-Pentachlorobiphenyl			123
3,3',4,4',5-Pentachlorobiphenyl	126[f]	126[f]	
2',3,3',4,4'-Hexachlorobiphenyl	128	128	
2,2',3,4,4',5'-Hexachlorobiphenyl	138	138	
2,2',3,5,5',6-Hexachlorobiphenyl			151
2,2',4,4',5,5'-Hexachlorobiphenyl	153	153	
2,3,3',4,4',5-Hexachlorobiphenyl		156	
2,3,3',4,4',5-Hexachlorobiphenyl			157
2,3,3',4,4',6-Hexachlorobiphenyl			158
2,3',4,4',5,5'-Hexachlorobiphenyl			167
2,3',4,4',5',6-Hexachlorobiphenyl			168

TABLE 8. (cont.)

PCB Congener[a]	Congener Number[b]		
	Summation[c]	Highest Priority[d]	Second Priority[e]
3,3',4,4',5,5'-Hexachlorobiphenyl	169[f]	169[f]	
2,2',3,3',4,4',5-Heptachlorobiphenyl	170	170	
2,2',3,4,4',5,5'-Heptachlorobiphenyl	180	180	
2,2',3,4,4',5',6-Heptachlorobiphenyl		183	
2,2',3,4,4',6,6'-Heptachlorobiphenyl		184	
2,2',3,4',5,5',6-Heptachlorobiphenyl	187		187
2,3,3',4,4',5,5' Heptachlorobiphenyl			189
2,2',3,3',4,4',5,6-Octachlorobiphenyl		195	
2,2',3,3',4,5,5',6'-Octachlorobiphenyl			201
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl		206	
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl		209	

Note: PCB - polychlorinated biphenyl

[a] PCB congeners recommended for quantitation, from dichlorobiphenyl through decachlorobiphenyl.

[b] Congeners are identified by their International Union of Pure and Applied Chemistry (IUPAC) number, as referenced in Ballschmiter and Zell (1980) and Mullin et al. (1984).

[c] These congeners are summed to determine total PCB concentration using the approach in NOAA (1989).

[d] PCB congeners having highest priority for potential environmental importance based on potential for toxicity, frequency of occurrence in environmental samples, and relative abundance in animal tissues (McFarland and Clarke 1989).

[e] PCB congeners having second priority for potential environmental importance based on potential for toxicity, frequency of occurrence in environmental samples, and relative abundance in animal tissues (McFarland and Clarke 1989).

[f] To separate PCBs 126 and 169, it is necessary to initially utilize an enrichment step with an activated carbon column (Smith 1981).

The overall toxicity of nonortho-substituted PCBs at a site can be assessed based on a comparison with the toxicity of 2,3,7,8-TCDD. A similar procedure can be used for assessing the toxicity of a mixture of dioxins and furans. In this "toxicity equivalency factor" (TEF) approach, potency values of individual congeners (relative to TCDD) and their respective sediment concentrations are used to derive a summed 2,3,7,8-TCDD equivalent (U.S. EPA 1989d; Table 9). EPA and the USACE are developing guidance on the use of this approach.

To ensure that contaminants not included in the list of target analytes are not overlooked in the chemical characterization of the dredged material, the analytical results should also be scrutinized by trained personnel. The presence of persistent unknown analytes should be noted. Methods involving GC/MS techniques for organic compounds are recommended for the identification of any unknown analytes.

2.8.3 Chemical Analysis of Water

Analysis to determine the potential release of dissolved contaminants from the dredged material (standard elutriate) may be necessary to make determinations of water column toxicity (see U.S. EPA and USACE 1994). Elutriate tests involve mixing dredged material with dredging site water and allowing the mixture to settle. The portion of the dredged material that is considered to have the potential to impact the water column is the supernatant remaining after undisturbed settling and centrifugation. Chemical analysis of the elutriate allows a direct comparison, after allowance for mixing, to applicable water quality standards. When collecting samples for elutriate testing, consideration should be given to the large volumes of water and sediment required to prepare replicate samples for analysis. In some instances, when there is poor settling, the elutriate preparation has to be performed successively several times to accumulate enough water for testing. The following sections discuss the selection of target analytes and techniques for water analyses. QA considerations are summarized in Section 2.10.5.

2.8.3.1 Selection of Target Analytes

Historical water quality information from the dredging site should be evaluated along with data obtained from the chemical analysis of sediment samples to select target analytes. Chemical evaluation of the dredged material provides a known list of contaminants that might affect the water column. All target analytes identified in the sediment should initially be considered potential targets for water analysis. Nonpriority pollutant chemical components which are found in measurable concentrations in the sediments should be included as target analytes if review of the literature indicates that these analytes have the

TABLE 9. Methodology for Toxicity Equivalency Factors

Because toxicity information on some dioxin and furan species is scarce, a structure-activity relationship has been assumed. The toxicity of each cogener is expressed as a fraction of the toxicity of 2,3,7,8 TCDD.

Compound	TEF
2,3,7,8 TCDD	1
other TCDD	0
2,3,7,8-PeCDDs	0.5
other PeCDDs	0
2,3,7,8-HxCDDs	0.1
other HxCDDs	0
2,3,7,8-HpCDDs	0.01
other HpCDDs	0
OCDD	0.001
2,3,7,8-TCDF	0.1
other TCDFs	0
1,2,3,7,8-PeCDF	0.05
2,3,4,7,8-PeCDF	0.5
other PeCDFs	0
2,3,7,8-HxCDFs	0.1
other HxCDFs	0
2,3,7,8-HpCDFs	0.01
other HpCDFs	0
OCDF	0.001

potential to bioaccumulate in animals (i.e., have a high K_{ow} or bioconcentration factor [BCF]) and/or are of toxicological concern) (Table 10).

2.8.3.2 Selection of Analytical Techniques

In contrast to freshwater, there generally are no EPA-approved methods for analysis of saline water although widely accepted methods have existed for some time (e.g., Strickland and Parsons 1972; Grasshof et al. 1983; Parsons et al. 1984). Application of the freshwater methods to saltwater will frequently result in higher detection limits than are common for freshwater unless care is taken to control the effects of salt on the analytical signal. Modifications or substitute methods (e.g., additional extract concentration steps, larger sample sizes, or concentration of extracts to smaller volumes) might be necessary to properly determine analyte concentrations in saltwater or to meet the desired TDLs. It is extremely important to ascertain a laboratory's ability to execute methods and attain acceptable TDLs in matrices containing up to 3 percent sodium chloride.

Once the list of target analytes for water has been established, analytical methods should be determined. The water volume required for specific analytical methods may vary. A minimum of 1 L of elutriate should be prepared for metals analysis (as little as 100 mL may be analyzed). One liter of elutriate should be analyzed for organic compounds. Sample size should also include the additional volume required for the matrix spike and matrix spike duplicate analyses, required for analysis of both metals and organic compounds. Sample size is one of the limiting factors in determining detection limits for water analyses, but TDLs below the water quality standard should be the goal in all cases. Participating laboratories should routinely report detection limits achieved for a given analyte.

Detailed methods for the analysis of organic and inorganic priority pollutants in water are referenced in 40 CFR 136 and in U.S. EPA (1983). Additional approved methods include U.S. EPA (1986a,b; 1988a,b,c; 1990d,e), APHA (1989), ASTM (1991a), and Tetra Tech (1985). Analysis of the semivolatile organic priority pollutants involves a solvent extraction of water with an optional sample cleanup procedure and analysis using GC or GC/MS. The volatile priority pollutants are determined by using purge-and-trap techniques and are analyzed by either GC or GC/MS. If dioxin (i.e., 2,3,7,8,-TCDD) analysis is necessary, Kuehl et al. (1987), Smith et al. (1984), U.S. EPA (1989b; Method 8290), or U.S. EPA (1990f; Method 1613) should be consulted. EPA Method 1613 is the recommended procedure for measuring the tetra- through octa-PCDDs and PCDFs.

A primary requirement for analysis of inorganic and organic priority pollutants is to obtain detection limits that will result in usable, quantitative data that can

TABLE 10. OCTANOL/WATER PARTITION COEFFICIENTS FOR ORGANIC COMPOUND PRIORITY POLLUTANTS AND 301(h) PESTICIDES

Pollutant	Octanol/Water Partition Coefficient (log K_{ow})	Pollutant	Octanol/Water Partition Coefficient (log K_{ow})
Di-*n*-octyl phthalate	9.2	Parathion[a]	3.8
Indeno[1,2,3-cd]pyrene	7.7	Chlorobenzene	3.8
Benzo[ghi]perylene	7.0	2,4,6-Trichlorophenol	3.7
PCB-1260	6.9	ß-Endosulfan	3.6
Mirex[a]	6.9	Endosulfan sulfate	3.6
Benzo[k]fluoranthene	6.8	α-Endosulfan	3.6
Benzo[b]fluoranthene	6.6	Naphthalene	3.6
PCB-1248	6.1	Fluorotrichloromethane[b]	3.5
2,3,7,8-TCDD (dioxin)	6.1	1,4-Dichlorobenzene	3.5
Benzo[a]pyrene	6.0	1,3-Dichlorobenzene	3.4
Chlordane	6.0	1,2-Dichlorobenzene	3.4
PCB-1242	6.0	Toxaphene	3.3
4,4'-DDD	6.0	Ethylbenzene	3.1
Dibenz[a,h]anthracene	6.0	*N*-Nitrosodiphenylamine	3.1
PCB-1016	5.9	*P*-Chloro-*m* cresol	3.1
4,4'-DDT	5.7	2,4-Dichlorophenol	3.1
4,4'-DDE	5.7	3,3'-Dichlorobenzene	3.0
Benz[a]anthracene	5.6	Aldrin	3.0
Chrysene	5.6	1,2-Diphenylhydrazine	2.9
Endrin aldehyde	5.6	4-Nitrophenol	2.9
Fluoranthene	5.5	Malathion[a]	2.9
Hexachlorocyclopentadiene	5.5	Tetrachloroethene	2.9
Dieldrin	5.5	4,6-Dinitro-*o*-cresol	2.8
Heptachlor	5.4	Tetrachloroethene	2.6
Heptachlor epoxide	5.4	Bis[2-chloroisopropyl]ether	2.6
Hexachlorobenzene	5.2	1,1,1-Trichloroethane	2.5
Di-*n*-butyl phthalate	5.1	Trichloroethene	2.4
4-Bromophenyl phenyl ether	5.1	2,4-Dimethylphenol	2.4
Pentachlorophenol	5.0	1,1,2,2-Tetrachloroethane	2.4
4-Chlorophenyl phenyl ether	4.9	Bromoform	2.3
Pyrene	4.9	1,2-Dichloropropane	2.3
2-Chloronaphthalene	4.7	Toluene	2.2
Endrin	4.6	1,1,2-Trichloroethane	2.2
PCB-1232	4.5	Guthion[a]	2.2
Phenanthrene	4.5	Dichlorodiflouromethane[b]	2.2
Fluorene	4.4	2-Chlorophenol	2.2
Anthracene	4.3	Benzene	2.1
Methoxychlor[a]	4.3	Chlorodibromomethane	2.1
Hexachlorobutadiene	4.3	2,4-Dinitrotoluene	2.1
1,2,4-Trichlorobenzene	4.2	2,6-Dinitrotoluene	2.0
Bis[2-ethylhexyl]phthalate	4.2	*trans*-1,2-Dichloropropene	2.0
Acenaphthylene	4.1	*cis*-1,3-Dichloropropene	2.0
Butyl benzyl phthalate	4.0	Demeton[a]	1.9
PCB-1221	4.0	Chloroform	1.9
Hexachloroethane	3.9	Dichlorobromomethane	1.9
Acenaphthene	3.9	Nitrobenzene	1.9
α-Hexachlorocyclohexane	3.8	Benzidine	1.8
δ-Hexachlorocyclohexane	3.8	1,1-Dichloroethane	1.8
ß-Hexachlorocyclohexane	3.8	2-Nitrophenol	1.8
γ-Hexachlorocyclohexane	3.8	Isophorone	1.7

TABLE 10. (cont.)

Pollutant	Octanol/Water Partition Coefficient (log K_{ow})	Pollutant	Octanol/Water Partition Coefficient (log K_{ow})
Dimethyl phthalate	1.6	2-Chloroethylvinylether	1.3
Chloroethane	1.5	Bis[2-chloroethoxy]methane	1.3
2,4-Dinitrophenol	1.5	Acrylonitrile	1.2
1,1-Dichloroethylene	1.5	Bis[2-chloroethyl]ether	1.1
Phenol	1.5	Bromomethane	1.0
1,2-Dichloroethane	1.4	Acrolein	0.9
Diethyl phthalate	1.4	Chloromethane	0.9
N-nitrosodipropylamine	1.3	Vinyl chloride	0.6
Dichloromethane	1.3	*N*-nitrosodimethylamine	0.6

Source: Tetra Tech (1985)

Note: Mixtures, such as PCB Aroclors ®, cannot have discrete K_{ow} values; however, the value given is a rough estimate for the mean. [It is recommended that all PCB analyses use congener-specific methods. All PCB congeners have a log $K_{ow} > 4$ (L. Burkhardt, EPA Duluth, pers. comm.).]

[a] 301(h) pesticides not on the priority pollutant list.

[b] No longer on priority pollutant or 301(h) list.

subsequently be compared against applicable water quality standards or criteria, as appropriate. Analysis of saline water for metals is subject to matrix interferences from salts, particularly sodium and chloride ions, when the samples are concentrated prior to instrumental analysis. The gold amalgamation method using cold-vapor atomic absorption spectrometry (CVAA) analysis is recommended to eliminate saline water matrix interferences for mercury analysis. Methods using solvent extraction and atomic absorption spectrometry analysis may be required to reduce saline water matrix interferences for other target metals. Other methods appropriate for metals include: cadmium, copper, lead, iron, zinc, silver (Danielson et al. 1978); arsenic (EPRI 1986); selenium and antimony (Sturgeon et al. 1985); low levels of mercury (Bloom et al. 1983); and tributyltin (Rice et al. 1987). GFAA techniques after extraction are recommended for the analysis of metals, with the exception of mercury. All PCB and pesticide analyses should be performed using GC/ECD methods because such analysis (e.g., NOAA 1989) will result in lower detection limits. PCBs should be quantified as specific congeners (Mullin et al. 1984; Stalling et al. 1987) and as total PCBs based on the summation of particular congeners (NOAA 1989).

2.8.4 Chemical Analysis of Tissue

This section discusses the selection of target analytes and techniques for tissue analyses. QA considerations are summarized in Section 2.10.6.

2.8.4.1 Selection of Target Analytes

Bioaccumulation is evaluated by analyzing tissues of test organisms for contaminants determined to be of concern for a specific dredged material. Sediment contaminant data and available information on the bioaccumulation potential of those analytes have to be interpreted to establish target analytes.

The *n*-octanol/water partition coefficient (K_{ow}) is used to estimate the BCFs of chemicals in organism/water systems (Chiou et al. 1977; Kenaga and Goring 1980; Veith et al. 1980; Mackay 1982). The potential for bioaccumulation generally increases as K_{ow} increases, particularly for compounds with log K_{ow} less than approximately 6. Above this value, there is less of a tendency for bioaccumulation potential to increase with increasing K_{ow}. Consequently, the relative potential for bioaccumulation of organic compounds can be estimated from the K_{ow} of the compounds. U.S. EPA (1985) recommends that compounds for which the log K_{ow} is greater than 3.5 be considered for further evaluation of bioaccumulation potential. The organic compound classes of priority pollutants with the greatest potential to bioaccumulate are PAHs, PCBs, pesticides, and some phthalate esters. Generally, the volatile organic, phenol, and organonitrogen priority pollutants are not readily bioaccumulated, but exceptions

include the chlorinated benzenes and the chlorinated phenols. Table 10 provides data for organic priority pollutants based on K_{ow}. Specific target analytes for PCBs and PAHs are discussed in Section 2.8.2. The water content and percent lipids in tissue should be routinely determined as a part of tissue analyses for organic contaminants.

Table 11 ranks the bioaccumulation potential of the inorganic priority pollutants based on calculated BCFs. Dredged material contaminants with BCFs greater than 1,000 (log BCF > 3) should be further evaluated for bioaccumulation potential.

Tables 10 and 11 should be used with caution because they are based on calculated bioconcentration from water. Sediment bioaccumulation tests, in contrast, are concerned with accumulation from a complex medium via all possible routes of uptake. The appropriate use of the tables is to help in selecting contaminants of concern for bioaccumulation analysis by providing a general indication of the relative potential for various chemicals to accumulate in tissues.

The strategy for selecting contaminants for tissue analysis should include three considerations:

- The target analyte is a contaminant of concern and is present in the sediment as determined by sediment chemical analyses
- The target analyte has a high potential to accumulate and persist in tissues
- The target analyte is of toxicological concern.

Contaminants with a lower potential to bioaccumulate, but which are present at high concentrations in the sediments, should also be included in the target list because bioavailability can increase with concentration. Conversely, contaminants with a high accumulation potential and of high toxicological concern should be considered as target analytes, even if they are only present at low concentrations in the sediments. Nonpriority-pollutant contaminants that are found in measurable concentrations in the sediments should be included as targets for tissue analysis if they have the potential to bioaccumulate and persist in tissues, and are of toxicological concern.

2.8.4.2 Selection of Analytical Techniques

At present, formally approved standard methods for the analysis of priority pollutants and other contaminants in tissues are not available. However, studies conducted for EPA and other agencies have developed analytical

TABLE 11. BIOCONCENTRATION FACTORS (BCF) OF INORGANIC PRIORITY POLLUTANTS

Inorganic Pollutant	Log BCF
Metals	
Methylmercury	4.6
Phenylmercury	4.6
Mercuric acetate	3.5
Copper	3.1
Zinc	2.8
Arsenic	2.5
Cadmium	2.5
Lead	2.2
Chromium IV	2.1
Chromium III	2.1
Mercury	2.0
Nickel	1.7
Thallium	1.2
Antimony	ND
Silver	ND
Selenium	ND
Beryllium	ND
Nonmetals	
Cyanide	ND
Asbestos	ND

Source: Tetra Tech (1986b)

Note: ND - no data

methods capable of identifying and quantifying most organic and inorganic priority pollutants in tissues. The amount of tissue required for analysis is dependent on the analytical procedure and the tissue moisture content. General guidance, but *not* firm recommendations, for the amount of tissue required is provided in Table 5. The required amounts may vary depending on the analytes, matrices, detection limits, and particular analytical laboratory. Tissue moisture content should be determined for each sample to enable data to be converted from a wet-weight to a dry-weight basis for some data users.

Detection limits depend on the sample size as well as the specific analytical procedure. Recommended TDLs for dredged material evaluations are provided in Section 2.3.2 (see Table 3). TDLs should be specified based on the intended use of the data and specific needs of each evaluation.

The recommended methods for the analysis of semivolatile organic pollutants are described in NOAA (1989). The procedure involves serial extraction of homogenized tissue samples with methylene chloride, followed by alumina and gel-permeation column cleanup procedures that remove co-extracted lipids. An automated gel-permeation procedure described by Sloan et al. (1993) is recommended for rapid, efficient, reproducible sample cleanup. The extract is concentrated and analyzed for semivolatile organic pollutants using GC with capillary fused-silica columns to achieve sufficient analyte resolution. If dioxin (i.e., 2,3,7,8-TCDD) analysis is being performed, the methods of Mehrle et al. (1988), Smith et al. (1984), Kuehl et al. (1987), U.S. EPA (1989b; Method 8290), or U.S. EPA (1990f; Method 1613) should be consulted. EPA Method 1613 is the recommended procedure for measuring the tetra- through octa-PCDDs and PCDFs.

Chlorinated hydrocarbons (e.g., PCBs and chlorinated pesticides) should be analyzed by GC/ECD. PCBs should be quantified as specific congeners (Mullin et al. 1984; Stalling et al. 1987) and not by industrial formulations (e.g., Aroclors®) because the levels of PCBs in tissues result from complex processes, including selective accumulation and metabolism (see the discussion of PCBs in Section 2.8.2.2). Lower detection limits and positive identification of PCBs and pesticides can be obtained by using chemical ionization mass spectrometry.

The same tissue extract is analyzed for other semivolatile pollutants (e.g., PAHs, phthalate esters, nitrosamines, phenols) using GC/MS as described by NOAA (1989), Battelle (1985), and Tetra Tech (1986b). These GC/MS methods are similar to EPA Method 8270 for solid wastes and soils (U.S. EPA 1986a). Lowest detection limits are achieved by operating the mass spectrometer in the selective ion monitoring mode. Decisions to perform analysis of nonchlorinated hydrocarbons and resulting data interpretation should consider that many of these analytes are readily metabolized by most fish and many

invertebrates. Analytical methods for analysis of tissue samples for volatile priority pollutants are found in Tetra Tech (1986b).

Tissue lipid content is of importance in the interpretation of bioaccumulation information. A lipid determination should be performed on all biota submitted for organic analysis if 1) food chain models will be used, 2) test organisms could spawn during the test, or 3) special circumstances occur, such as those requiring risk assessment. Bligh and Dyer (1959) provide an acceptable method, and the various available methods are evaluated by Randall et al. (1991).

Analysis for priority pollutant metals involves a nitric acid or nitric acid/perchloric acid digestion of the tissue sample and subsequent analysis of the acid extract using atomic absorption spectrometry or ICP techniques. Procedures in Tetra Tech (1986b) are generally recommended. NOAA (1989) methods may also be used and are recommended when low detection levels are required. Microwave technology may be used for tissue digestion to reduce contamination and to improve recovery of metals (Nakashima et al. 1988). This methodology is consistent with tissue analyses performed by NOAA (1989), except for the microwave heating steps. Mercury analysis requires the use of CVAA methods (U.S. EPA 1991c). The matrix interferences encountered in analysis of metals in tissue may require case-specific techniques for overcoming interference problems. If tributyltin analysis is being performed, the methods of Rice et al. (1987), NCASI (1986), or Uhler et al. (1989) should be consulted.

2.9 *DATA VALIDATION, REDUCTION, AND REPORTING*

This section describes procedures for data compilation and verification prior to being accepted for making technical conclusions. In addition, special equations may be required and used to make calculations, models may be used in data analysis, criteria may be used to validate the integrity of data that support final conclusions, and methods may be used to identify and treat data that may not be representative of environmental conditions.

The following specific information should be included in the QA project plan:

- The principal criteria that will be used to validate data integrity during collection and reporting of data (the criteria selected will depend on the level of validation required to meet the data quality objectives)
- The data reduction scheme planned for collected data, including all equations used to calculate the concentration or value of the measured parameter and reporting units

- The methods used to identify and treat outliers (i.e., data that fall outside the upper and lower limits such as ±3 standard deviations of the mean value) and nondetectable data
- The data flow or reporting scheme from collection of original data through storage of validated concentrations (a flowchart is usually necessary)
- Statistical formulas and sample calculations planned for collected data
- Key individuals who will handle the data in this reporting scheme.

QC procedures designed to eliminate errors during the mathematical and/or statistical reduction of data should also be included in the QA project plan. QC in data processing may include both manual and automated review. Input data should be checked and verified to confirm compatibility and to flag outliers for confirmation (i.e., verify that data are outliers and not data for highly contaminated sediment, water, or tissue). Computerized data plots can be routinely used as a tool for rapid identification of outliers that can then be verified using standard statistical procedures.

2.9.1 Data Validation

Once the laboratory has completed the requested sample analyses, the analytical results are compiled, printed out, and submitted as a data package, which has been signed by the laboratory's project manager. This package may include computer disks, magnetic tape, or other forms of electronically stored information. Data packages may range in size from a few pages to several cartons of documents, depending on the nature and extent of the analyses performed. The cost of this documentation can vary from no charge (in cases where only the final results of an analysis are reported) to hundreds of dollars over the cost of reporting only the final results of an analysis.

The data and information collected during the dredged material evaluation should be carefully reviewed as to their relevancy, completeness, and quality. The data must be relevant to the overall objective of the project. Data quality should be verified by comparing reported detection limits and QC results to TDLs and QC limits, respectively, specified for the current dredged material evaluation.

As soon as new data packages are received from the laboratory, they should be checked for completeness and data usability and, ideally, dated and duplicated. Dating is important for establishing the laboratory's adherence to schedules identified in the statement of work. Duplication assures that a clean reference copy is always kept on file. Checking each element of the data

package for completeness of information, precision of analytical methods, and bias of all measurements helps to determine whether acceptable data from each type of analysis have been supplied by the laboratory.

Screening for data quality requires knowledge of the sample holding times and conditions, the types of analyses requested, and the form in which data were to be delivered by the laboratory. Review of the statement of work is essential to determine any special conditions or requests that may have been stated at the onset of the analyses. Recommended lists of laboratory deliverables for different types of chemical analyses are provided in Tables 1 and 2. This initial screening of data can be performed by appropriate staff or the project manager.

Data validation, or the process of assessing data quality, can begin after determining that the data package is complete. Analytical laboratories strive to produce data that conform to the requested statement of work, and they typically perform internal checks to assure that the data meet a standard level of quality. However, data validation is an independent check on laboratory performance and is intended to assure that quality of reported data meets the needs identified in the QA project plan.

Data validation involves all procedures used to accept or reject data after collection and prior to use. These include screening, editing, verifying, and reviewing through external performance evaluation audits. Data validation procedures ensure that objectives for data precision and bias were met, that data were generated in accordance with the QA project plan and standard operating procedures, and that data are traceable and defensible. All chemical data should be reported with their associated analytical sensitivity, precision, and bias. In addition, the quantification level achieved by the laboratory should be compared to specific TDLs.

The QA project plan should also specify an appropriate level of data validation for the intended data use. Examples of four alternative levels of validation effort for chemical data are summarized in Table 12. These four data validation levels range from complete, 100-percent review of the data package (Level 1) to acceptance of the data package without any evaluation (Level 4).

The QA project plan should also specify who will perform the evaluations called for in Levels 1, 2, or 3. The following options should be considered for chemical data:

- Perform a brief assessment and rely on specialists to resolve outstanding concerns. This assessment is equivalent to Level 3 (Table 12).
- Perform a complete review for Level 1 or 2 using qualified staff and technical guidelines for QA specialists (see Footnote a in Table 12).

TABLE 12. LEVELS OF DATA VALIDATION

Level 1	100 percent of the data (including data for laboratory quality control samples) are independently validated using the data quality objectives established for the project. Calculations and the possibility of transcription errors are checked. Instrument performance and original data for the analytical standards used to calibrate the method are evaluated to ensure that the values reported for detection limits and data values are appropriate. The bias and precision of the data are calculated and a summary of corrections and data quality is prepared.[a]
Level 2	20 percent of the sample data and 100 percent of the laboratory quality control samples are validated. Except for the lower level of effort in checking data for samples, the same checks conducted in Level 1 are performed. If transcription errors or other concerns (e.g., correct identification of chemicals in the samples) are found in the initial check on field samples, then data for an additional 10–20 percent of the samples should be reviewed. If numerous errors are found, then the entire data package should be reviewed.
Level 3	Only the summary results of the laboratory analyses are evaluated. The data values are assumed to be correctly reported by the laboratory. Data quality is assessed by comparing summary data reported by the laboratory for blanks, bias, precision, and detection limits with data quality objectives in the QA project plan. No checks on the calibration of the method are performed, other than comparing the laboratory's summary of calibrations with limits specified in the QA project plan.
Level 4	No additional validation of the data is performed. The internal reviews performed by the laboratory are judged adequate for the project.

[a] Screening checks that can be easily performed by the project manager are provided in (U.S. EPA 1991d). Step-by-step procedures used by quality assurance specialists to validate data for analyses of organic compounds and metals can be found in EPA's functional guidelines for data review (U.S. EPA 1988a,b). These guidelines were developed for analyses conducted according to the statements of work for EPA's Contract Laboratory Program and are updated periodically. Regional interpretation of these detailed procedures is also contained in *Data Validation Guidance Manual for Selected Sediment Variables* (PTI 1989b), a draft report released by the Washington Department of Ecology's Sediment Management Unit in June 1989. A simplified version of this guidance is provided in *Data Quality Evaluation for Proposed Dredged Material Disposal Projects* (PTI 1989a), another report released by the Sediment Management Unit in June 1989.

- Send the data package to an outside technical specialist for review, specifying either Level 1, 2, or 3.

Providing instructions for conducting a thorough technical validation of chemical data is beyond the scope of this document. Examples of detailed technical guidance of this nature can be found in a pair of publications, *Laboratory Data Validation: Functional Guidelines for Evaluating Inorganics Analyses* (U.S. EPA 1988a) and *Laboratory Data Validation: Functional Guidelines for Evaluating Organics Analyses* (U.S. EPA 1988b). Examples of simple evaluations that can be conducted by a project manager are also provided in U.S. EPA (1991d). The evaluation criteria in Figure 1 (abstracted from U.S. EPA [1991d]) provide several signs that should alert a project manager to potential problems with data acceptability.

2.9.2 Data Reduction and Reporting

The QA project plan should summarize how validated data will be analyzed to reach conclusions, including major tools that will be used for statistical evaluations. In this section, a flow chart is useful to show the reduction of original laboratory data to final tabulated data in the project report. A summary should also be provided of the major kinds of data analyses that will be conducted (e.g., health risk assessments, mapping of chemical distributions). In addition, the format, content, and distribution of any data reports for the project should be summarized.

2.10 INTERNAL QUALITY CONTROL CHECKS

The various control samples that will be used internally by the laboratory or sample collection team to assess quality are described in this section of the QA project plan. For most environmental investigations, 10–30 percent of all samples may be analyzed specifically for purposes of quality control. In some special cases (e.g., when the number of samples is small and the need to establish the validity of analytical data is large), as many as 50 percent of all samples are used for this purpose. These QC samples may be used to check the bias and precision of the overall analytical system and to evaluate the performances of individual analytical instruments or the technicians that operate them.

In addition to calibration procedures described in Section 2.7, this section of the guidance document (and Appendix C) summarizes the most widely used QC samples as follows:

- Blanks

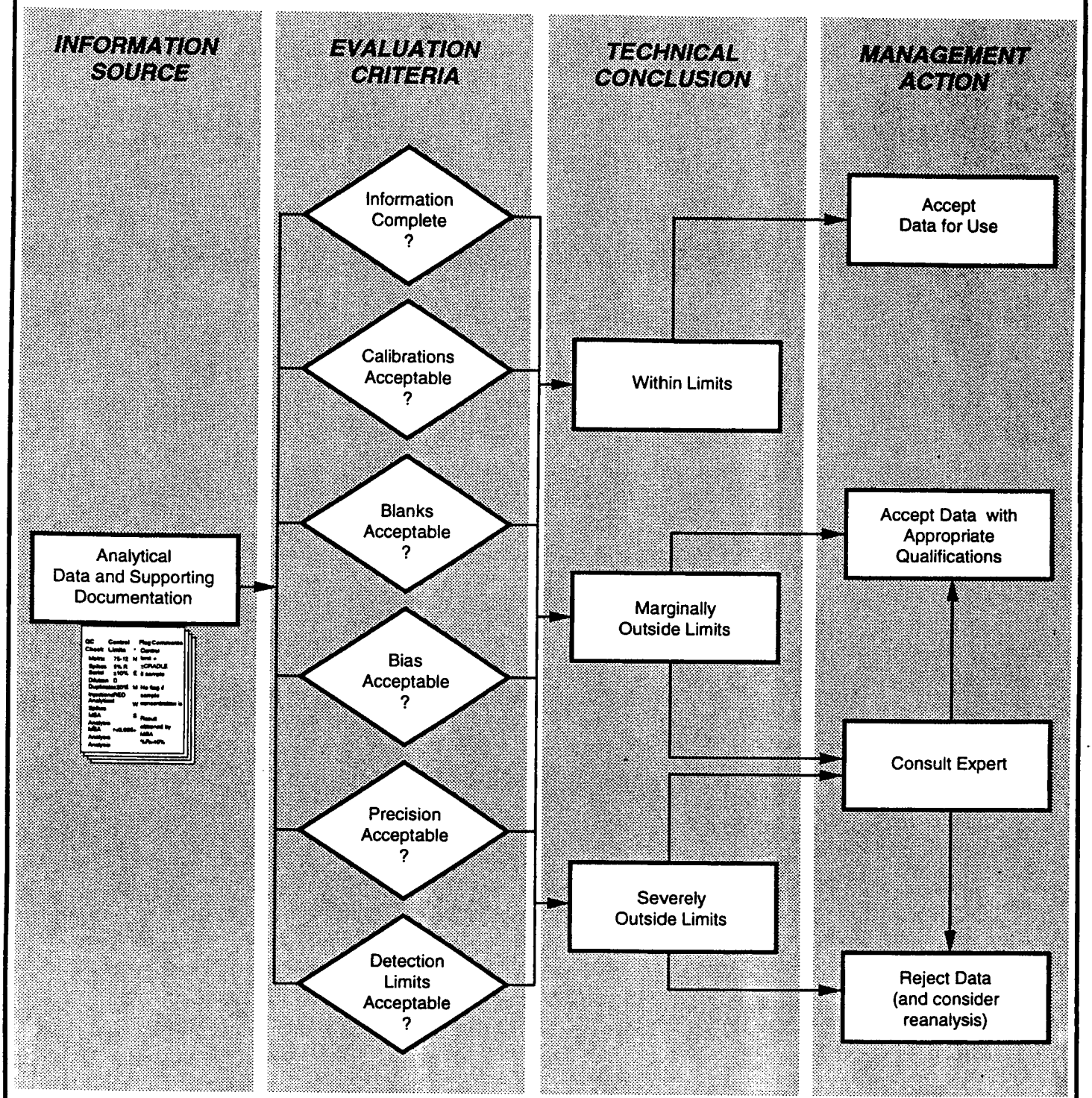

Figure 1. Guidance for data assessment and screening for data quality.

- Matrix spike samples
- Surrogate spike compounds
- Check standards, including:
  - Spiked method blanks
  - Laboratory control samples
  - Reference materials
- Matrix replicates (split in the laboratory from one field sample)
- Field replicates (collected as separate field samples from one location).

QC procedures for sediment, water, and tissue analyses are discussed in more detail in the following sections. Field QC results are not used to qualify data, but only to help support conclusions arrived at by the review of the entire data set.

The government authorities for the program may require that certain samples be submitted on a routine basis to government laboratories for analysis, and EPA or USACE may participate in some studies. These activities provide an independent QA check on activities being performed and on data being generated and are discussed in Section 2.11 (*Performance and System Audits*).

2.10.1 Priority and Frequency of Quality Control Checks

Which QC samples will be used in analyses should be determined during project planning. The frequency of QC procedures is dependent upon the type of analysis and the objectives of the project (as established in Section 2.3). The statements of work for EPA's CLP (U.S. EPA 1990d,e) specify the types of checks to be used during sample analysis. Determining the actual numbers of samples and how often they must be used is also a part of this process. These specifications, called QC sample frequencies, represent the minimum levels of effort for a project. Increasing the frequency of QC samples may be an appropriate measure when the expected concentrations of chemicals are close to the detection limit, when data on low chemical concentrations are needed, when there is a suspected problem with the laboratory, or when existing data indicate elevated chemical concentrations such that removal or other actions may be required. In such cases, the need for increased precision may justify the cost of extra QC samples.

The relative importance, rationale, and relative frequency of calibration and each kind of QC sample are discussed in Appendix C. The following priority, rationale, and frequency of use is recommended for each procedure:

1. **Method blank samples** are one of the highest priority checks on QC, because they provide an assessment of possible laboratory contamination (and the means to correct results for such contamination), and are used to determine the detection limit. As a result, method blank analyses are always required; at least one analysis is usually performed for each group of samples that are processed by a laboratory. In contrast, the need for other kinds of blank samples (bottle, transport, or field equipment) is usually project-specific and depends on the likelihood of contamination from solvents, reagents, and instruments used in the project; the matrix being analyzed; or the contaminants of concern. A bottle blank consists of an unopened empty sampling bottle that is prepared and retained in the field laboratory. A trip travel blank consists of deionized water and preservative (as added to the samples) that is prepared in the laboratory and transported to the sampling site. A field or decontamination blank consists of deionized water from the sample collection device and preservative (as added to the samples) that is prepared at the sampling site.

2. **Matrix spike samples** are high-priority checks on QC and should always be analyzed to indicate the bias of analytical measurements due to interfering substances or matrix effects. The suggested frequency is 1 matrix spike for every 20 samples analyzed. If more than 1 matrix type is present (e.g., samples containing primarily sand and samples containing primarily of silt within the same group), then each matrix type should be spiked at the suggested frequency. Duplicate matrix spike samples analyzed at a frequency of 1 duplicate for every 20 samples can serve as an acceptable means of indicating both the bias and precision of measurement for a particular sample. Duplicate matrix spike samples may provide the only information on precision for contaminants that are rarely detected in samples.

3. **Surrogate spike compounds** are high-priority checks on QC that are used to evaluate analytical recovery (e.g., sample extraction efficiency) of organic compounds of interest from individual samples. Surrogate spike compounds should be added to every sample, including blanks and matrix spike samples, prior to performing sample processing, to monitor extraction efficiency on a sample-by-sample basis. This kind of check is only used when the identity of the surrogate compound can be reasonably confirmed (e.g., by mass spectroscopy). Because a surrogate compound is

chemically similar to the associated compound of interest and is added to the sample in a known amount, its known recovery is indicative of that of the compound of interest.

Variations in recovery that can be seen using surrogate spike compounds with each sample will not necessarily be reflected in duplicate matrix spike analyses conducted on only a few of the samples. The reasons for possible differences between surrogate spike analyses and matrix spike analyses relate to sample heterogeneity and how these QC samples are prepared. For example, matrix spike analyses provide an indication of chemical recovery for the general sample matrix tested. However, this matrix may differ among individual samples leading to a range of recoveries for surrogate spike compounds among samples. In addition, surrogate spike compounds are often added at a lower concentration than matrix spike compounds. This difference in spiking concentration sometimes results in reasonable recovery of the higher-concentration matrix spike compounds but poorer recovery of the lower-concentration surrogate spike compounds. Finally, matrix spike compounds are typically identical to compounds of interest in the samples, while surrogate spike compounds are usually selected because they are not present in environmental samples, but still mimic the behavior of compounds of interest. Therefore, there can be more uncertainty in quantifying the recovery of matrix spike compounds (after subtracting the estimated concentration of the compounds of interest in the sample) than the recovery of surrogate spike compounds.

4. **Check standards** should be used whenever available as a high-priority check on laboratory performance. Check standards include laboratory control samples, reference materials prepared by an independent testing facility, and spiked method blanks prepared by the laboratory. By comparing the results of check standards with those of sample-specific measurements (e.g., matrix spike samples and surrogate compound recovery), an overall assessment of bias and precision can be obtained. The laboratory should be contacted prior to analysis to determine what laboratory control samples can be used. Catalogues from organizations such as National Institute for Standards and Technology and the National Research Council of Canada are available that list reference materials for different sediment, water, and tissue samples (see Section 2.11.2).

   Reference materials provide a standardized basis for comparison among laboratories or between different rounds of analysis at one laboratory. Therefore, reference materials should always be used

when comparison of results with other projects is an intended data use. At least 1 analysis of a reference material for every 20 samples is recommended for this purpose. Similarly, spiked method blanks should be used as acceptable checks on laboratory performance whenever a new procedure is used or when laboratories with no established track record for a standard or nonstandard procedure will be performing the analysis.

5. **Analytical replicate samples** should be included as a medium-priority check on laboratory precision. Analytical replicate samples better indicate the precision of measurements on actual samples than do matrix spike duplicates because the contaminants have been incorporated into the sample by environmental processes rather than having been spiked in a laboratory setting. The suggested frequency is 1 replicate sample for every 20 samples for each matrix type analyzed. For organic analyses, analysis of analytical spike duplicate samples are sometimes a higher priority than matrix replicate samples if budgets are limited. The reason for this preference is because many organic compounds of interest may not be present in samples unless they are added as spiked compounds.

6. **Field replicate samples** should be included if measuring sampling variability is a critical component of the study design. Otherwise, collection of field replicate samples is discretionary and a lower priority than the other QC samples. Field replicate samples should be submitted to the laboratory as blind samples. When included, the suggested frequency is at least 1 field replicate for every 20 samples analyzed. One of the field replicate samples should also be split by the laboratory into analytical duplicates so that both laboratory and laboratory-plus-sampling variability can be determined on the same sample. By obtaining both measures on the same sample, the influence of sampling variability can be better discerned. It is possible that analytical variability can mask sampling variability at a location.

2.10.2 Specifying Quality Control Limits

Prior to performing a chemical analysis, recognized limits on analytical performance should be established. These limits are established largely through the analysis of QC samples. QC limits apply to all internal QC checks for field measurements, physical characterizations, bioaccumulation studies, and toxicity tests. Many laboratories have established limits that are applicable to their own measurement systems. These limits should be evaluated to ensure that they are at least as stringent as general guidelines or that the reasons for a less

stringent limit are acceptable. Also, if a laboratory has consistently demonstrated better performance than indicated by general guidelines, limits tied to this better performance should be used to indicate when there may be a problem at that laboratory. For example, if surrogate recoveries for benzene in sediment samples have consistently been between 85 and 105 percent, a recovery of 70 percent indicates an analytical problem that should be investigated even if the general guideline for acceptable recovery is 50 percent. It may be useful to establish different kinds of limits when working with laboratories. For example, the following two kinds of limits are used by PSEP (1990c) and are similar to limits used in EPA's CLP.

Warning limits are values indicating that data from the analysis of QC samples should be qualified (e.g., that they represent estimated or questionable values) before they can be relied upon in a project. These limits serve to warn the project staff that the analytical system, instrument, or method may not be performing normally and that data should be qualified as "estimated" before using the results for technical analysis. The standard value for warning limits are ±2 times the standard deviation (U.S. EPA 1979). Examples of warning limits used by the Puget Sound Estuary Program are provided in Table 13. Such limits provide a means of ensuring that reported data are consistently qualified, an important consideration when combining data in a regional database.

If necessary to meet project goals, project managers may specify warning limits as more stringent contractual requirements in laboratory statements of work. For example, Puget Sound Estuary Program guidelines for organic compound analyses state that the warning limits for the minimum recovery of surrogate spike and matrix spike compounds are 50 percent of the amount added prior to sample extraction. Data that do not meet this minimum requirement would normally be qualified as estimates. However, the project manager could apply more stringent criteria and decide to reject data that do not meet warning limits, which would require reanalysis of the samples associated with those QC samples that do not meet these limits. These more stringent criteria are termed control limits.

Control limits are limits placed on the acceptability of data from the analysis of QC samples. Exceedance of control limits informs the analyst and the project manager that the analytical system or instrument is performing abnormally and needs to be corrected. Control limits should be contractually binding on laboratories, and statements of work should provide the project manager or designee with sole discretion in enforcing the limits. Data obtained under these circumstances should be corrected before they are resubmitted by the laboratory. Data that exceed control limits are often rejected and excluded from a project database, although there may be special circumstances that warrant acceptance of the data as estimated values. The reasons for making such an

TABLE 13. EXAMPLE WARNING AND CONTROL LIMITS FOR CALIBRATION AND QUALITY CONTROL SAMPLES[a]

Analysis Type	Recommended Warning Limit	Recommended Control Limit
Ongoing calibration	Project manager decision[b]	> ±25 percent of the average response measured in the initial calibration
Surrogate spikes	< 50 percent recovery[c]	Follow EPA Contract Laboratory Program guidelines
Method blanks	Exceeds the TDL	Exceeds 5 times the TDL
Reference materials	95 percent confidence interval, if certified	To be determined
Matrix spikes	50–150 percent recovery	To be determined[d]
Spiked method blanks (check standards)	50–150 percent recovery	To be determined
Matrix replicates	35 percent coefficient of variation	> ±50 percent coefficient of variation (or a factor of 2 for duplicates)
Field replicates	Project manager decision	Project manager decision

Note: TDL - target detection limit

[a] Warning and control limits used in the Puget Sound Estuary Program for the analysis of organic compounds (PSEP 1990c).

[b] See U.S. EPA (1991d) for specific examples of project manager decisions for warning or control limits.

[c] Except when using the isotope dilution technique.

[d] Zero percent spike recovery requires rejection of data.

exception should always be documented in a QA report for the data (see Appendix F).

Unlike warning limits, control limits and appropriate corrective actions (such as instrument recalibration, elimination of sources of laboratory contamination, or sample reanalysis) should be clearly identified in the statement of work. The standard value for control limits are ±3 times the standard deviation (U.S. EPA 1979). Examples of regional control limits used by the Puget Sound Estuary Program are also provided in Table 13. In those cases that require a project manager's decision to determine the appropriate control limit, it is recommended that the associated warning limit be used as an control limit to produce data that will have broad applicability (including use in enforcement proceedings). Control limits should be enforced with discretion because some environmental samples are inherently difficult to analyze. Recommended actions under different circumstances are provided below.

2.10.3 *Quality Control Considerations for Physical Analysis of Sediments*

The procedures used for the physical analysis of sediments should include a QC component. QC procedures for grain size analysis and total solids/specific gravity determinations are necessary to ensure that the data meet acceptable criteria for precision and bias. To measure precision, triplicate analyses should be performed for every 20 samples analyzed. TOC is a special case, where all samples should be analyzed in triplicate, as recommended by the analytical method. In addition, 1 procedural blank per 20 samples should be run, and the results reported for TOC analysis. Standards used for TOC determinations should be verified by independent check standards to confirm the bias of the results. Quality control limits should be agreed upon for each analytical procedure, and should be consistent with the overall QA project plan.

2.10.4 *Quality Control Considerations for Chemical Analysis of Sediments*

Methods for the chemical analysis of priority pollutants in sediments should include detailed QC procedures and requirements that should be followed rigorously throughout the evaluation. General procedures include the analysis of a procedural blank, a matrix duplicate, a matrix spike along with every 10–20 samples processed, and surrogate spike compounds. All analytical instruments should be calibrated at least daily (see Section 2.7.1). All calibration data should be submitted to the laboratory project QA coordinator for review. The QA/QC program should document the ability of the selected methods to address the high salt content of sediments from marine and estuarine areas.

Analytical precision can be measured by analyzing 1 sample in duplicate or triplicate for every 10–20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported; however, if triplicates are analyzed, the percent relative standard deviation should be reported.

2.10.5 *Quality Control Considerations for Chemical Analysis of Water*

Methods recommended for the chemical analysis of priority pollutants in water include detailed QC procedures and requirements that should be followed closely throughout the evaluations. General procedures should include the analysis of a procedural blank, a matrix duplicate, a matrix spike for every 10–20 samples processed, and surrogate spike compounds (for organic analyses only). Analytical precision can be measured by analyzing 1 sample in triplicate or duplicate for every 10–20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported; however, if triplicates are analyzed, the percent relative standard deviation should be reported. Analytical bias can be measured by analyzing SRM, a matrix containing a known amount of a pure reagent. Recoveries of surrogate spikes and matrix spikes should be used to measure for precision and bias; results from these analyses should be well documented. Special quality control is required for ICP and GC/MS analyses. Initial calibrations using three or five standards (varying in concentration) are required for analyses of inorganic and organic compounds, respectively, before analyzing samples (see Section 2.7.2). Subsequent calibration checks should be performed for every 10–20 samples analyzed.

2.10.6 *Quality Control Considerations for Chemical Analysis of Tissue*

Methods recommended for the chemical analysis of priority pollutants in tissue include detailed QC procedures and requirements that should be followed closely throughout the evaluations. General procedures should include the analysis of a procedural blank, a matrix duplicate, a matrix spike for every 10–20 samples processed, and surrogate spike compounds (for organic analyses only). Analytical precision can be measured by analyzing 1 sample in triplicate or duplicate for every 10–20 samples analyzed. If duplicates are analyzed, the relative percent difference should be reported; however, if triplicates are analyzed, the percent relative standard deviation should be reported. Analytical bias can be measured with the appropriate SRMs. Precision and bias determinations should be performed with the same frequency as the blanks and matrix spikes.

2.11 PERFORMANCE AND SYSTEM AUDITS

Procedures to determine the effectiveness of the QC program and its implementation are summarized in this section of the QA project plan. Each QA project plan should describe the various audits required to monitor the capability and performance of all measurement systems. Audits include a careful evaluation of both field and laboratory QC procedures. They are an essential part of the field and laboratory QA program and consist of two basic types: performance audits and system audits. For example, analyses of performance evaluation samples may simply be used for comparison with the results of independent laboratories (a form of performance audit), or comprehensive audits may be conducted by the government of the entire field or laboratory operations (a system audit).

Performance and system audits should be conducted by individuals not directly involved in the measurement process. A performance auditor independently collects data using performance evaluation samples, field blanks, trip blanks, duplicate samples, and spiked samples. Performance audits may be conducted soon after the measurement systems begin generating data. They may be repeated periodically as required by task needs, duration, and cost. U.S. EPA (1991e) should be reviewed for auditing the performance of laboratories performing aquatic toxicity tests.

A systems audit consists of a review of the total data production process. It includes onsite reviews of field and laboratory operational systems. EPA and/or USACE will develop and conduct external system audits based on the approved project plan. An example of a systems audit checklist is provided in Appendices A and G.

2.11.1 Procedures for Pre-Award Inspections of Laboratories

The pre-award inspection is a kind of system audit for assessing the laboratory's overall capabilities. This assessment includes a determination that the laboratory personnel are appropriately qualified and that the required equipment is available and is adequately maintained. It establishes the groundwork necessary to ensure that tests will be conducted properly, provides the initial contact between government and laboratory staff, and emphasizes the importance that government places on quality work and products.

The purpose of the pre-award inspection is to verify the following:

- The laboratory has an independent QA/QC program
- Written work plans are available for each test that describe the approach to be used in storing, handling, and analyzing samples

- Technically sound, written standard operating procedures are available for all study activities
- Qualifications and training of staff are appropriate and documented
- All equipment is properly calibrated and maintained
- Approved analytical procedures are being followed.

2.11.2 Interlaboratory Comparisons

It is important that data collected and processed at various laboratories be comparable. As part of the performance audit process, laboratories may be required to participate in analysis of performance evaluation samples related to specific projects. In particular, laboratory proficiency should be demonstrated before a laboratory negotiates a contract and yearly thereafter. Each laboratory participating in a proficiency test is required to analyze samples prepared to a known concentration. Analytes used in preparation of the samples should originate from a recognized source of SRMs such as the National Institute for Standards and Technology. Proficiency testing programs already established by the government may be used (e.g., EPA Environmental Monitoring and Systems Laboratory scoring system), or a program may be designed specifically for dredged material evaluations.

In addition, the performance evaluation samples prepared by EPA Environmental Monitoring and Systems Laboratory (Las Vegas, Nevada) for the CLP may be used to assess interlaboratory comparability. Analytical results are compared with predetermined criteria of acceptability (e.g., values that fall within the 95 percent confidence interval are considered acceptable). The QA project plan should indicate, where applicable, scheduled participation in all interlaboratory calibration exercises.

Reference materials are substances with well-characterized properties that are useful for assessing the bias of an analysis and auditing analytical performances among laboratories. SRMs are certified reference materials containing precise concentrations of chemicals, accurately determined by a variety of technically valid procedures, and are issued by the National Institute of Standards and Technology. Currently, SRMs are not available for the physical measurements or all pollutants in sediments; however, where possible, available SRMs or other regional reference materials that have been repeatedly tested should be analyzed with every 20 samples processed.

SRMs for most organic compounds are not currently available for seawater, but reference materials for many inorganic chemicals may be obtained from the organizations listed in Table 14. Seawater matrix spikes of target analytes (e.g., seawater spiked with National Institute for Standards and Technology

TABLE 14. SOURCES OF STANDARD REFERENCE MATERIALS

PCBs		
National Research Council of Canada	Marine sediment	HS–1 and HS–2
PAHs		
National Research Council of Canada	Marine sediment	HS–3, HS–4, HS–5, HS–6
National Institute for Standards and Technology	Sediment	SRM #1647 and SRM #1597
Metals		
National Bureau of Standards	Estuarine sediment	SRM #1646
National Research Council of Canada	Marine sediment	MESS–1, BCSS–1, PACS–1
	Dogfish liver	DOLT-1
	Dogfish muscle	DORM-1
	Lobster hepatopancreas	TORT-1
International Atomic Energy Agency	Marine sediment	SD–N–1/2(TM)
	Fish flesh	MA-A-2(TM)
	Mussel tissue	MAL-1(TM)

Standard reference materials (SRMs) may be obtained from the following organizations:

Organic Constituents

U.S. Department of Commerce
National Institute for Standards and Technology
Office of Standard Reference Materials
Room B3111 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301) 975–6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Atlantic Research Laboratory
1411 Oxford Street
Halifax, Nova Scotia, Canada B3H 3Z1
Telephone: (902) 426–8280

Inorganic Constituents

U.S. Department of Commerce
National Institute for Standards and Technology
Office of Standard Reference Materials
Room B3111 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301) 975–6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Division of Chemistry
Montreal Road
Ottawa, Ontario, Canada K1A 0R9
Telephone: (613) 993–2359

SRM 1647 for PAH) should be used to check analytical bias. Some available SRMs for priority pollutant metals in seawater are National Research Council of Canada seawater CASS–1 and seawater NASS–2.

SRMs for organic priority pollutants in tissues are currently not available. The National Institute of Standards and Technology is presently developing SRMs for organic analytes. Tissue matrix spikes of target analytes should be used to fulfill analytical accuracy requirements for organic analyses.

Because new SRMs appear constantly, current listings of appropriate agencies should be consulted frequently. SRMs that are readily available and commonly used are included in Table 14.

2.11.3 Routine System Audits

Routine system audits during the technical evaluation ensure that laboratories are complying with the QA project plan. It is suggested that checklists be developed for reviewing training records, equipment specifications, QC procedures for analytical tasks, management organization, etc. The government should also establish laboratory review files for quick assessment of the laboratory's activity on a study, and to aid in monitoring the overall quality of the work. Procedures for external system audits by the government are similar to the internal systems audits conducted by the laboratories themselves.

2.12 FACILITIES

The QA Project Plan should provide a complete, detailed description of the physical layout of the laboratory, define space for each test area, describe traffic-flow patterns, and document special laboratory needs. The design and layout of laboratory facilities are important to maintain sample integrity and prevent cross-contamination. The specific areas to be used for the various evaluations should be identified. Aspects of the dredging study that warrant separate facilities include the following:

- Receiving
- Sample storage
- Sample preparation
- Sample testing
- Reagent storage
- Data reduction and analysis.

2.13 PREVENTIVE MAINTENANCE

Procedures for maintaining field and laboratory equipment in a ready state are described in this section, including identification of critical spare parts that must be available to ensure that data completeness will not be jeopardized by equipment failure. Regular servicing must be implemented and documented.

The QA project plan should describe how field and laboratory equipment essential to sample collection and analysis will be maintained in proper working order. Preventive maintenance may be in the form of: 1) scheduled maintenance activities to minimize costly downtime and ensure accuracy of measurement systems, and 2) available spare parts, backup systems, and equipment. Equipment should be subject to regular inspection and preventive maintenance procedures to ensure proper working order. Instruments should have periodic calibration and preventive maintenance performed by qualified technical personnel, and a permanent record should be kept of calibrations, problems diagnosed, and corrective actions applied. An acceptance testing program for key materials used in the performance of environmental measurements (chemical and biological materials) should be applied prior to their use.

2.14 CALCULATION OF DATA QUALITY INDICATORS

Specific equations or procedures used to assess the precision, bias, and completeness of the data are identified in this section.

The calculations and equations used routinely in QA review (e.g., relative percent difference of duplicates) as well as the type of samples (e.g., blanks, replicates) analyzed to assess precision, bias, and completeness of the data must be presented in the QA project plan. Routine procedures for measuring precision and bias include the use of replicate analyses, SRMs, and matrix spikes. The following routine procedures can be used to measure precision and bias:

1. **Replicate analysis**

   Precision for duplicate chemical analyses will be calculated as the relative percent difference:

$$\text{Relative percent difference} = \frac{\text{abs}[D_1 - D_2]}{(D_1 + D_2)/2} \times 100$$

   where:

   D_1 = sample value

D_2 = duplicate sample value
abs = absolute value.

Precision for the replicate will be calculated as the relative standard deviation:

$$\text{Percent relative standard deviation} = \frac{\sigma}{x} \times 100$$

where:

x = mean of three or more results
σ = standard deviation of three or more results.

$$\sigma = \left[\frac{\sum (x-x)^2}{n-1}\right]^{1/2}$$

2. Matrix and surrogate spikes

Bias of these measurements will be calculated as the ratio of the measured value to the known spiked quantity:

$$\text{Percent recovery} = \frac{\text{spiked result} - \text{unspiked result}}{\text{spike added}} \times 100$$

3. Method blank

Method blank results are assessed to determine the existence and magnitude of contamination. Guidelines for evaluating blank results and specific actions to be taken are identified in U.S. EPA (1988a,b). Sample results will not be corrected by subtracting a blank value.

4. Laboratory control sample

Bias of these measurements will be calculated as the ratio of the measured value to the referenced value:

$$\text{Percent recovery} = \frac{\text{measured value}}{\text{referenced value}} \times 100$$

5. Completeness

Completeness will be measured for each set of data received by dividing the number of valid (i.e., accepted) measurements actually obtained by the number of measurements that were planned:

$$\text{Completeness} = \frac{\text{valid data points obtained}}{\text{total data points planned}} \times 100$$

To be considered complete, the data set should also contain all QC check analyses that verify the accuracy (precision and bias) of the results.

2.15 CORRECTIVE ACTIONS

Major problems that could arise during field or laboratory operations, predetermined corrective actions for these problems, and the individual responsible for each corrective action are identified in this section.

One purpose of any QA program is to identify nonconformance as quickly as possible. A nonconformance event is defined as any event that does not follow defined methods, procedures, or protocols, or any occurrence that may affect the quality of the data or study. A QA program should have a corrective action plan and should provide feedback to appropriate management authority defining how all nonconformance events were addressed and corrected.

Corrective actions fall into two categories: 1) handling of analytical or equipment malfunctions, and 2) handling of nonconformance or noncompliance with the QA requirements that have been established. During field and laboratory operations, the supervisor is responsible for correcting equipment malfunctions. All corrective measures taken should be documented (e.g., a written standard operating procedure for the corrective action) and, if required, an alteration checklist should be completed.

Corrective action procedures should be described for each project and include the following elements:

- Procedures for corrective actions when predetermined limits for data acceptability are exceeded (see DQO discussion in Section 2.3)
- For each measurement system, the individual responsible for initiating the corrective action and the individual responsible for approving the corrective action.

Corrective actions for field procedures should be described in a separate section from the corrective actions that would apply to the data or laboratory analysis. Corrective actions may be initiated as a result of other QA activities including performance audits, system audits, interlaboratory/interfield comparison studies, and QA program audits. An example of a corrective actions checklist is provided in Appendix A.

2.16 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The process of assuring data quality does not end with the data review. A report summarizing the sampling event (see Appendix H) and the QA review of the analytical data package should be prepared, samples should be properly stored or disposed of, and laboratory data should be archived in a storage file or database. Technical interpretation of the data begins after the QA review has been completed. Once data interpretation is complete, the results of the project should be carefully examined to determine how closely the original project goals and objectives were met. QA reviews are particularly useful for providing data users with a written record of data concerns and a documented rationale for why certain data were accepted as estimates or were rejected.

QA project plans provide a mechanism for periodic reporting to management on the performance of measurement systems and data quality. At a minimum, these reports should include:

- Periodic assessment of measurement data accuracy (precision and bias) and completeness
- Results of performance and system audits
- Significant QA problems and recommended solutions.

The individuals responsible for preparing the periodic reports should be identified. The final report for each project should include a separate QA section that summarizes data quality information contained in the periodic reports. These reports may be prepared by the project manager if a brief evaluation was conducted, or by QA specialists if a detailed review was requested by the project manager.

2.16.1 Preparing Basic Quality Assurance Reports

Basic QA reports should summarize all conclusions concerning data acceptability and should note significant QA problems that were found. The table of contents for a basic QA report should include the following:

- **Data summary**—The data summary section should discuss the number of samples collected, the laboratory(s) that analyzed the samples, and a summary of the data that were qualified during the QA review.
- **Holding times**—The holding time section should briefly discuss the holding time requirements and holding time exceedances.

- **Analytical methods**—The analytical methods section should briefly describe the methods of analysis, any departures from the methods, and any calibration or instrument-specific QC criteria exceedances.

- **Accuracy**—The accuracy section should include a discussion of QC criteria and exceedances for 1) analytical bias (surrogate compound, laboratory control sample, matrix spike, and reference material recoveries) and 2) precision of matrix replicates (and matrix spike duplicates for organic compounds.

- **Method blanks**—The method blank section should include a brief discussion of method blank QC criteria and exceedances.

QA reviews are usually included as appendices to technical project reports. In any case, the QA review becomes part of the documented project file, which also includes the original data package and any computer files used in data compilation and analysis.

2.16.2 Preparing Detailed Quality Assurance Reports

Depending on the project objectives, a more detailed QA report may be desired. An example of a detailed QA review for a metals data package is provided in Appendix F. In addition to the sections outlined for the basic QA report, the detailed QA report should also include:

- **Introduction**—The introduction should give a brief overview of the purpose of data collection and brief summaries of how the samples were collected and processed in the field.

- **Sample set description**—The sample set section should describe the number of samples sent to each laboratory, including the number of field blanks, field replicates, SRMs, and interlaboratory split samples.

- **Sample delivery group description**—The sample delivery group section should briefly describe how the samples were sorted by the analytical laboratories (how many sample delivery groups were returned by the laboratory), and whether or not the QC criteria were performed at the correct frequency for each sample delivery group.

- **Field QC summary**—The field QC section should discuss the evaluation of the field blank and replicate results for the sample survey.

- **Interlaboratory comparison**—The interlaboratory section, where applicable, should describe the evaluation of the split samples as compared to the corresponding samples analyzed by the contract laboratory.
- **Field results description**—The field results section, where applicable, should present tabular summaries of all data with appropriate qualifiers.

For organic analyses, a discussion of the results of instrument tuning (if applicable), instrument calibration analyses, internal standard performance (if applicable), and summation of any factors that could effect overall data quality (e.g., system degradation) should also be included in the detailed QA report.

2.17 REFERENCES

References cited in the QA project plan should be provided at the end of the plan.

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4. GLOSSARY

Accuracy — The ability to obtain precisely a nonbiased (true) value. Accuracy as used in this document is the combined measure of precision and bias (see footnote at beginning of Section 2).

Acid Volatile Sulfide — The sulfides removed from sediment by cold acid extraction, consisting mainly of H_2S and FeS. AVS is a possible predictive tool for divalent metal sediment toxicity.

Analyte — The specific component measured in a chemical analysis.

Bias — Deviation of the measurement from the true value. Usually expressed as the percent recovery of a known amount of a chemical added to a sample at the start of a chemical analysis. Bias (along with precision) is a component of the overall accuracy of a system.

Bioaccumulation — The accumulation of contaminants in the tissue of organisms through any route, including respiration, ingestion, or direct contact with contaminated water, sediment, pore water, or dredged material.

Bioassay — A bioassay is a test using a biological system. It involves exposing an organism to a test material and determining a response. There are two major types of bioassays differentiated by response: **toxicity tests** which measure an effect (e.g., acute toxicity, sublethal/chronic toxicity) and **bioaccumulation tests** which measure a phenomenon (e.g., the uptake of contaminants into tissues).

Bioconcentration Factor — The degree to which an organism uptakes a substance from water.

Blanks	QC samples that are processed with the samples but contain only reagents. They are used to obtain the response of an analysis in the absence of a sample, including assessment of contamination from sources external to the sample.
Calibration	The systematic determination of the relationship of the response of the measurement system to the concentration of the analyte of interest. Instrument calibration performed before any samples are analyzed is called the **initial calibration**. Subsequent checks on the instrument calibration performed throughout the analyses of samples are called **continuing calibration**.
Chromatography	The process of selectively separating a mixture into its component compounds. The compounds are measured and presented graphically in the form of a **chromatogram** and digitally as a **quantification report**.
Cleanup	The process of removing certain components from sample extracts, performed to improve instrument sensitivity
Comparability	Reflects the confidence with which one data set can be compared with others and the expression of results consistent with other organizations reporting similar data. Comparability of analytical procedures also implies using analytical methodologies that produce results comparable in terms of precision, bias, and effective range of calibration.
Completeness	A measure of the amount of valid data *obtained* vs. the amount of data originally *intended* to be collected.
Confined Disposal Facility	A diked area, either in-water or upland, used to contain dredged material.
Contaminant	A chemical or biological substance in a form that can be incorporated into, onto, or be ingested by and that harms aquatic organisms, consumers of aquatic organisms, or users of the aquatic environment, and includes but is not limited to the

substances on the 307(a)(1) list of toxic pollutants promulgated on January 31, 1978 (43 *Federal Register* 4109).

Control Limit

A value for data from the analysis of QC checks indicating that a system or a method is not performing normally and that an appropriate corrective action should be taken. When control limits are exceeded, analyses should be halted; samples analyzed since the last QC sample may need reanalysis.

Control Sediment

A sediment used to confirm the biological acceptability of the test conditions and to help verify the health of the organisms during the test. Control sediment is essentially free of contaminants and compatible with the biological needs of the test organisms such that it has no discernable influence on the response being measured in the test. Test procedures are conducted with the control sediment in the same way as the reference sediment and dredged material. Control sediment may be the sediment from which the test organisms are collected or a laboratory sediment. Excessive mortality in the control sediment indicates a problem with the test conditions or organisms, and can invalidate the results of the corresponding dredged material test.

Data Package

The results of chemical analyses completed by a laboratory, compiled, printed out, and presented to the agency or individual requesting the analyses. The data package should include chromatograms, calculations, and tuning and calibration summaries, where appropriate. Also included in the data package may be computer disks, magnetic tape, or other forms of electronically stored data.

Data Quality Indicators

Surrogate spike recoveries, matrix spike recoveries, analytical values obtained for blanks, standard reference material, and performance evaluation samples for each parameter in each matrix.

Data Quality Objectives (DQOs)

Qualitative and quantitative statements of the overall uncertainty that a decision maker is willing to accept in results or decisions derived from

	environmental data. DQOs provide the framework for planning environmental data operations consistent with the data user's needs.
Detector	A device used in conjunction with an analytical instrument to determine the components of a sample.
Digestion	A process used prior to analysis that breaks down samples using acids (or bases). The end product is called a digestate. Other chemicals, called **matrix modifiers**, may be added to improve the final digestate.
Disposal Site	That portion of inland or ocean where specific disposal activities are permitted. It consists of a bottom surface area and any overlying volume of water.
Dredged Material	Material excavated or dredged from waters of the United States. A general discussion of the nature of dredged material is provided by Engler et al. (1991).
Dredged Material Discharge	Any addition of dredged material into waters of the United States, including: open water discharges; discharges from unconfined disposal operations (such as beach nourishment or other beneficial uses); discharges from confined disposal facilities which enter waters of the United States (such as effluent, surface runoff, or leachate); and overflow from dredge hoppers, scows, or other transport vessels.
Elutriate	Material prepared from the sediment dilution water and used for chemical analyses and toxicity testing.
Evaluation	The process of judging data in order to reach a decision.
Extraction	A chemical or mechanical procedure to remove semivolatile organic compounds from a sample matrix. The end product of extraction is called an **extract**.
Interference	Unwanted elements or compounds in a sample

	that have properties similar to those of the chemical of interest and that collectively cause unacceptable levels of bias in the results of a measurement or in sensitive measurements. Unless removed by an appropriate cleanup procedure, the interferant is carried along with the chemical of interest through the analytical procedure.
Ion	An atom or group of atoms that carries a positive or negative electric charge as a result of having lost or gained one or more electrons.
Matrix	The sample material (e.g., water, sediment, tissue) in which the chemicals of interest are found. Matrix refers to the physical structure of a sample and how chemicals are bound within this structure. At a gross level, tissue is one kind of sample matrix and soil is another. At a finer level, a sediment sample of silty sand containing large amounts of calcium carbonate from the shells of aquatic organisms represents a different sample matrix than a sediment sample of clayey silt containing a large amount of organic carbon from decaying vegetation.
Matrix Effects	Matrix effects are physical or chemical interactions between the sample material and the chemical of interest that can bias chemical measurements in either a negative or positive direction. Because matrix effects can vary from sample to sample and are often not well understood, they are a major source of variability in chemical analyses.
Matrix Spike Samples	QC check samples created by adding known amounts of chemicals of interest to actual samples, usually prior to extraction or digestion. Analysis of matrix spikes and matrix spike duplicates will provide an indication of bias due to matrix effects and an estimation of the precision of the results.
Metals	A group of naturally occurring elements. Certain metals (such as mercury, lead, nickel, zinc, and cadmium) can be of environmental concern when they are released to the environment in unnaturally high amounts. This group usually includes the metalloid arsenic.

Organic Compounds	Carbon-based substances commonly produced by animals or plants. **Organic chemicals** are chemical compounds based on carbon chains or rings and also containing hydrogen with or without oxygen, nitrogen, or other elements. Organic chemicals may be produced naturally by plants and animals or processed artificially using various chemical reactions.
Performance Audit	Audit of a laboratory's performance by testing a standard reference material. The test results are evaluated by the auditor.
Precision	The ability to replicate a value; the degree to which observations or measurements of the same property, usually obtained under similar conditions, conform to themselves. Usually expressed as standard deviation, variance, or range. Precision, along with bias, is a component of the overall accuracy of a system.
Quality Assurance	The total integrated program for assuring the reliability of data. A system for integrating the quality planning, quality control, quality assessment, and quality improvement efforts to meet user requirements and defined standards of quality with a stated level of confidence.
Quality Assurance Management Plan	A detailed document specifying guidelines and procedures to assure data quality at the program level (i.e., multiple projects).
Quality Assurance Project Plan	A detailed, project-specific document specifying guidelines and procedures to assure data quality during data collection, analysis, and reporting.
Quality Control	The overall system of technical activities for obtaining prescribed standards of performance in the monitoring and measurement process to meet user requirements.
Quality Control Checks	Blanks, replicates, and other samples used to assess the overall analytical system and to evaluate the performances of individual analytical instruments or the technicians that operate them.

Reference Materials — Materials or substances with well-characterized properties that are useful for assessing the accuracy of an analysis and comparing analytical performances among laboratories.

Reference Sediment — A sediment that serves as a point of comparison to identify potential effects of contaminants in the dredged material (see *Inland* and *Ocean Testing* manuals for further discussion).

Replicates — One of several identical samples. When two separate samples are taken from the same station, or when one sample is split into two separate samples and analyzed, these samples are called **duplicates**. When three identical samples are analyzed, these samples are called **triplicates**.

Representativeness — The degree to which sample data depict an existing environmental condition; a measure of the total variability associated with sampling and measuring that includes the two major error components: systematic error (bias) and random error. Sampling representativeness is accomplished through proper selection of sampling locations and sampling techniques, and collection of sufficient number of samples.

Sediment — Material, such as sand, silt, or clay, suspended in or settled on the bottom of a water body. The term *dredged material* refers to material which has been dredged from a water body (see definition of dredged material), while the term *sediment* refers to material in a water body prior to the dredging process.

Semivolatile Organic Compound — An organic compound with moderate vapor pressure that can be extracted from samples using organic solvents and analyzed by gas chromatography.

Spectrometry — The use of spectrographic techniques for deriving the physical constants of materials. Four basic forms of spectrometry commonly used are atomic absorption spectrometry (AA), inductively coupled plasma-atomic emission spectrometry (ICP) for metals, and ultraviolet spectrometry (UV) and

	fluorescence emission or excitation spectrometry for organic compounds.
Spiked Method Blanks	Method blanks to which known amounts of surrogate compounds and analytes have been spiked. Such samples are useful to verify acceptable method performance prior to and during routine analysis of samples containing organic compounds. Also known as check standards in some methods; independently prepared standards used to check for bias and to estimate the precision of measurements.
Standard Operating Procedure	A written document which details an operation, analysis, or action whose mechanisms are thoroughly prescribed and which is commonly accepted as the method for performing certain routine or repetitive tasks.
Standard Reference Material	Standard reference materials are certified reference materials containing precise concentrations of chemicals, accurately determined by a variety of technically valid procedures.
Statement of Work	A contract addendum used as a legally binding agreement between the individual or organization requesting an analysis and the individual, laboratory, or organization performing the actual tasks.
Surrogate Spike Compounds	Compounds with characteristics similar to those of compounds of interest that are added to a sample prior to extraction. They are used to estimate the recovery of organic compounds in a sample.
Target Detection Limit (TDL)	A performance goal set by consensus between the lowest, technically feasible, detection limit for routine analytical methods and available regulatory criteria or guidelines for evaluating dredged material. The TDL is, therefore, equal to or greater than the lowest amount of a chemical that can be reliably detected based on the variability of the blank response of routine analytical methods. However, the reliability of a chemical measurement generally increases as the concentration increases. Analytical costs may also be lower at higher detection limits. For these reasons, a TDL is

	typically set at not less than 10 times lower than available dredged material guidelines for potential biological effects associated with sediment chemical contamination.
Tests/Testing	Specific procedures which generate biological, chemical, and/or physical data to be used in evaluations. The data are usually quantitative but may be qualitative (e.g., taste, odor, organism behavior).
Toxicity Test	A **bioassay** which measures an effect (e.g., acute toxicity, sublethal/chronic toxicity). Not a **bioaccumulation test** (see definition of **bioassay**).
Volatile Organic Compound	An organic compound with a high vapor pressure that tends to evaporate readily from a sample.
Warning Limit	A value indicating that data from the analysis of QC checks are subject to qualification before they can be used in a project. When two or more sequential QC results fall outside of the warning limits, a systematic problem is indicated.
Water Quality Standard	A law or regulation that consists of the beneficial designated use or uses of a water body, the numeric and narrative water quality criteria that are necessary to protect the use or uses of that particular water body, and an anti-degradation statement.