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ENVIRONMENTAL SERVICES

Puget Sound Dredged Disposal Analysis Guidance Manual

Data Quality Evaluation for Proposed Dredged Material Disposal Projects

(QA-1)

For

Washington Department of Ecology
Olympia, Washington

June 1989

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PUGET SOUND DREDGED DISPOSAL ANALYSIS GUIDANCE MANUAL

DATA QUALITY EVALUATION FOR PROPOSED DREDGED MATERIAL DISPOSAL PROJECTS

For

Washington Department of Ecology
Sediment Management Unit
Mail Stop PV-11
Olympia, Washington 98504-8711

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1. INTRODUCTION

This manual provides guidance for the review of data submitted to the U.S. Army Corps of Engineers, Seattle District (Corps) by applicants for dredging permits. This data review process is termed QA1. The purpose of QA1, as described in the Puget Sound Dredged Disposal Analysis (PSDDA) management plan (PSDDA 1988a), is to establish if data are acceptable for determining the suitability of sediments for unconfined, open-water disposal. The handling, organization, and synthesis of sediment data described here are designed to streamline the data review process. The goal of QA1 is to ensure that sediment data from proposed projects have received adequate quality assurance (QA) review prior to a determination of suitability. To meet this need, field and laboratory QA information relevant to PSDDA data review is compiled by the field teams and analytical laboratories responsible for sample collection and testing.

The process of reviewing chemical and biological data to determine if they are suitable for incorporation in the sediment quality values database is termed QA2. QA2 is described in detail in the *Data Validation Guidance Manual for Selected Sediment Variables* (PTI 1989). QA2 will follow QA1 to establish if the data are acceptable for incorporation into the sediment quality values database maintained by the Washington Department of Ecology (Ecology).

1.1. BACKGROUND

PSDDA data review represents only one element of the PSDDA evaluation procedures. These procedures include sampling, testing, and test interpretation (i.e., against disposal guidelines) of dredged material proposed for unconfined, open-water disposal in Puget Sound [Evaluations Procedure Technical Appendix (PSDDA 1988b)]. These procedures were developed by an interagency committee, the Environmental Procedures Work Group (EPWG), composed of representatives from each of the PSDDA agencies: Ecology, Corps, U.S. Environmental Protection Agency (EPA) Region 10, and Washington Department of Natural Resources (DNR). Representatives from other federal and state agencies, Puget Sound ports, Indian tribes, and the public also assisted EPWG.

The goal of PSDDA is to provide publicly acceptable guidelines for environmentally safe, unconfined, open-water disposal of dredged material, and to provide Puget Sound-wide consistency and predictability in decisions concerning dredged material disposal. The PSDDA study was conducted in two phases addressing two geographic regions in Puget Sound. Phase I, which began in April 1985, was conducted over a 3½-year period and involved the central portion of Puget Sound from Tacoma to Everett. Phase II, which will be completed in late 1989, overlaps the Phase I schedule and includes the remainder of Puget Sound.

An understanding of the PSDDA evaluation procedures is needed prior to the description of the QA1 review process because sampling schemes, analytical protocols, and ultimate use of data influence the information generated, reviewed, and interpreted for a particular dredging project.

1.2. OVERALL QA PERSPECTIVE

The sequence of major QA activities associated with a project is summarized in Figure 1. Each project can be divided into four phases: 1) project planning, 2) data collection, 3) data quality review, and 4) data use.

1.2.1. Project Planning

The project planning phase contains two major QA elements: selection of analytical laboratories and development of a QA project plan (QAPP). The selection of an analytical laboratory is based on analytical capabilities (i.e., does the laboratory analyze for the variables of interest, are analytical techniques consistent with PSDDA requirements?), the laboratory's willingness to supply program-required information (e.g., QA1 checklists, required documentation), the ability of the laboratory to deliver results in a timely fashion, analytical and related costs, and convenience of laboratory location.

The QAPP defines all QA elements specific to a particular project. Most essential components of a QAPP have already been specified during PSDDA procedures development (PSDDA 1988b), either directly as part of the procedures development (e.g., sampling strategy, which is a component of sampling procedures) or by reference [e.g., specification of the Puget Sound Estuary Program (PSEP) sampling and analysis protocols (PSEP 1986)]. The PSDDA model QA plan (in preparation) will provide more detailed guidance. This QA1 report is intended as a control manual, which will be incorporated into the final users manual (probably as an appendix). This report lists the elements that will be included in the model QA plan. On completion of the model QA plan, QAPP will be required of all dredging applicants. The PSDDA model QA plan can be incorporated into QAPP by reference. Key elements of the PSDDA model QA plan will include the following:

- **Program description**—Outlines the scope of the program.
- **Program organization and responsibilities**—Identifies the overall organization of PSDDA, the relationship of QAPP within the overall program organization, and responsibilities of QA personnel in implementing QAPP.
- **Quality assurance objectives**—Identifies the PSDDA management objectives for accomplishing the scope of work addressed in the program description, including quantitative objectives for generating and measuring data in terms of accuracy, precision, completeness, representativeness, and comparability.
- **Sampling procedures**—Describes, for each measurement variable, the technique or guideline used to select sampling sites and specific sampling procedures. Provides charts, flow diagrams, or tables that delineate sampling operations (by example), containers and special conditions for the preparation of sampling equipment, and sampling preservation methods and holding times.
- **Sample chain-of-custody**—Identifies specific procedures to document sample control during transportation and storage, including preparation, identification, and appropriate labeling of sample containers and reagents, preservatives, or sampling media (e.g., filters). Also describes preparation and verification of signed chain-of-custody records that document sampling location, date, time, and type of medium; relinquishment of samples by signature, time, and date through each state of transfer

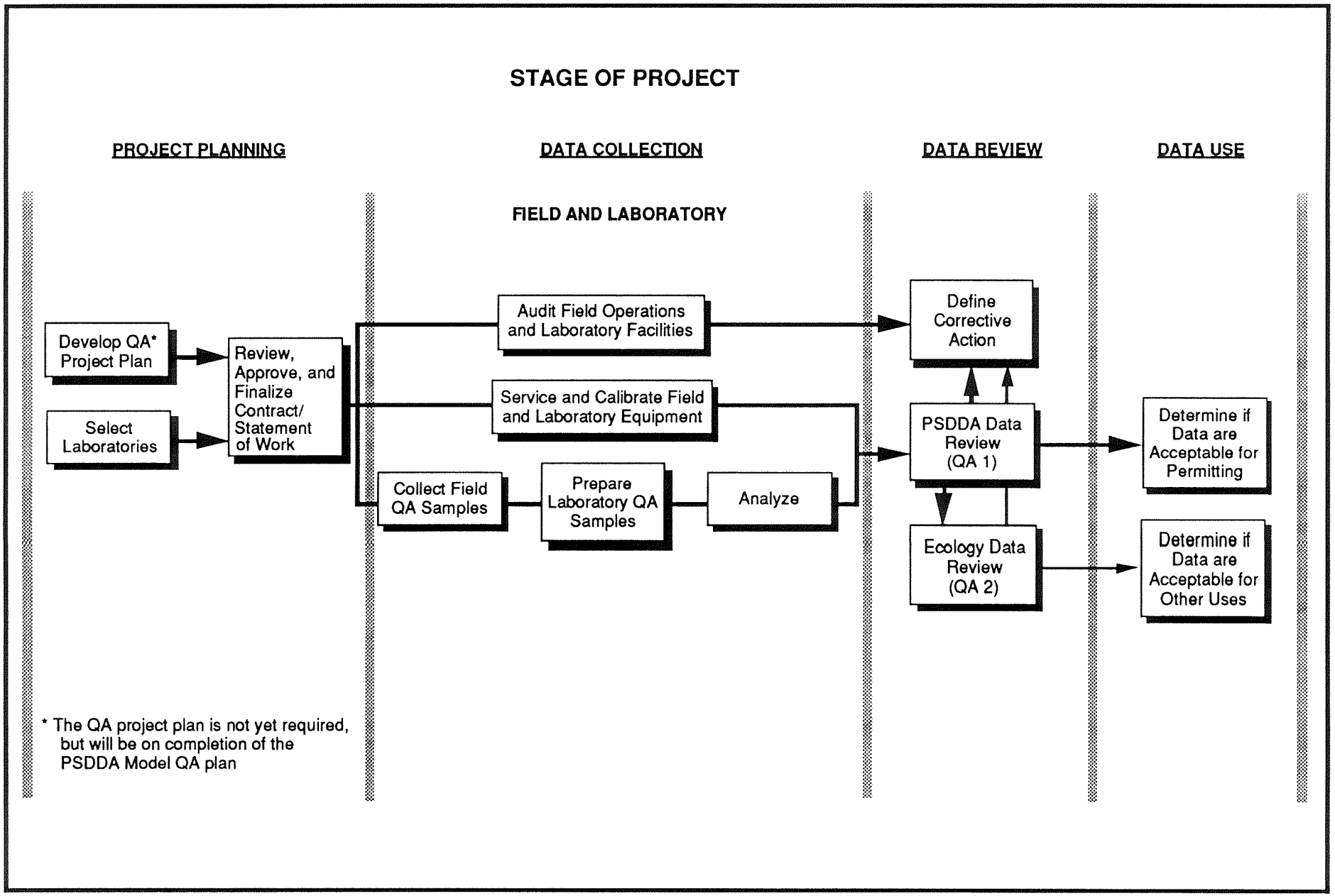

Figure 1. Overall Quality Assurance Perspective

to the laboratory; and control and identification of samples through the analytical process (including provisions for archiving unused portions of samples).

- **Calibration procedures, references, and frequencies**—Identifies procedures for properly maintaining the accuracy and precision of field and laboratory equipment, and for properly obtaining, using, and storing analytical standards.
- **Analytical procedures**—Identifies standard procedures for sample analysis by reference, and describes specialized procedures in detail.
- **Data reduction, validation, and reporting**—Provides the data reduction scheme for measurement data, including equations used for calculations, criteria used to validate data integrity, and methods used to identify and treat abnormal data or statistical outliers.
- **Internal quality control checks and frequency**—Identifies all procedures used to assess quality during sample collection and analysis, including the use and frequency of replicates, spikes, blanks, surrogate samples or reference materials, control charts, and calibration materials.
- **Quality assurance audits**—Describes procedures used to determine the effectiveness of the QA program and its implementation, including recommended system audits to ensure proper selection and use of measurement systems (e.g., by conducting onsite reviews in the field and laboratory), and recommended performance audits to ensure the proper continued generation of data (e.g., by implementing performance evaluation and round-robin studies, and reviewing analyses of blind and known reference materials).
- **Preventative maintenance procedures and schedules**—Details procedures for maintaining equipment in a ready state, including lists of critical spare parts.
- **Procedures and deliverables for data validation**—Provides a compilation of routine data analysis techniques used to assess data precision and accuracy, representativeness, comparability, and completeness of the measured parameters.
- **Corrective action**—Identifies predetermined limits for data acceptability beyond which corrective action is necessary, the specific corrective action to be taken for out-of-control data (including action in response to system and performance audits), and the individual responsible for each corrective action.

According to PSEP protocols, certain corrective actions should be performed by the laboratory during sample analysis. For example, matrix spike recoveries and analytical precision exceeding control limits should be investigated and, if possible, corrected by the laboratory during sample analysis. Because not all laboratories routinely monitor their performance on an ongoing basis, laboratory corrective actions should be specified in their contract. Often corrective actions are not specified to the laboratory, and are not followed, resulting in qualification or rejection of data during QA review conducted outside the laboratory.

1.2.2. Data Collection

Data collection, the second phase of the project, includes sample collection and sample analysis. Both field and laboratory activities require periodic audits to ensure protocols are followed. PSDDA recommends but does not require that a dredger conduct these audits of

laboratory and field activities. The timing and frequency of both laboratory and field audits are commonly specified in the QAPP. QA samples for the PSDDA program are generated and analyzed in the laboratory. Maintenance, service, and calibration of field and laboratory equipment are also essential components of the QA process, and records of these activities should be reviewed during audits.

PSDDA sampling addresses either the full or partial characterization of a potential dredging site. Full characterization (FC) of dredged material includes sampling, compositing, and testing as a basis for regulatory decisions on the acceptability of the material for unconfined, open-water disposal. For relatively large projects, the dredger may elect to perform partial characterization (PC) of sediments in the proposed dredging area if the dredger feels the project area is overranked. The PC is based on chemical analysis of a limited number of samples. Guidelines for FC and PC are provided in PSDDA (1988b). Information from PC may also be used to delete certain chemicals of concern from subsequent analyses after discussion of data and agreement of PSDDA agencies.

1.2.3. Data Quality Review

The major data characteristics reviewed during QA1 review include completeness, format, holding conditions, and QA sample results (e.g., matrix spikes, blanks). The general approach to PSDDA data review is described in Section 1.3. QA2, discussed in a separate document (PTI 1989), will include PSDDA QA data review elements as well as an additional measure of laboratory performance.

QA review requires a considerable amount of documentation from the laboratory (see Appendix A for documentation requirements). Complete documentation must be confirmed upon receipt of the laboratory's data package.

1.2.4. Data Use

Sediment data generated by dredgers serve many purposes. The quality of the data, in part, determines how they can be used. For example, QA1 determines if the data are acceptable for use as one factor in a permit decision process. QA2 determines the acceptability of the data for incorporation into the sediment quality values database. This database can be used to refine PSDDA screening level (SL) and maximum level (ML).

1.3. GENERAL QA1 APPROACH

Major steps leading to QA1 data review are summarized in Figure 2. During field sampling, a checklist is prepared by the dredger or the dredger's contractor. Included in this checklist are the area designation, sampling scheme, sampling and analytical strategy, and information on departures from protocols. During laboratory analysis, chemistry and bioassay checklists are prepared that summarize the key QA elements to be evaluated for QA1. Individual chemistry checklists are prepared for each data type (i.e., conventional variables, metals, semivolatile organic compounds, volatile organic compounds). Checklists are also prepared for individual bioassays (i.e., amphipod mortality, juvenile infauna mortality, sediment larval abnormality (solid and suspended phase), and Microtox™. Bioaccumulation data are summarized on similar checklists. These checklists include information on completeness, holding times, data format (e.g., significant figures, units), and QA samples (e.g., blanks, replicates).

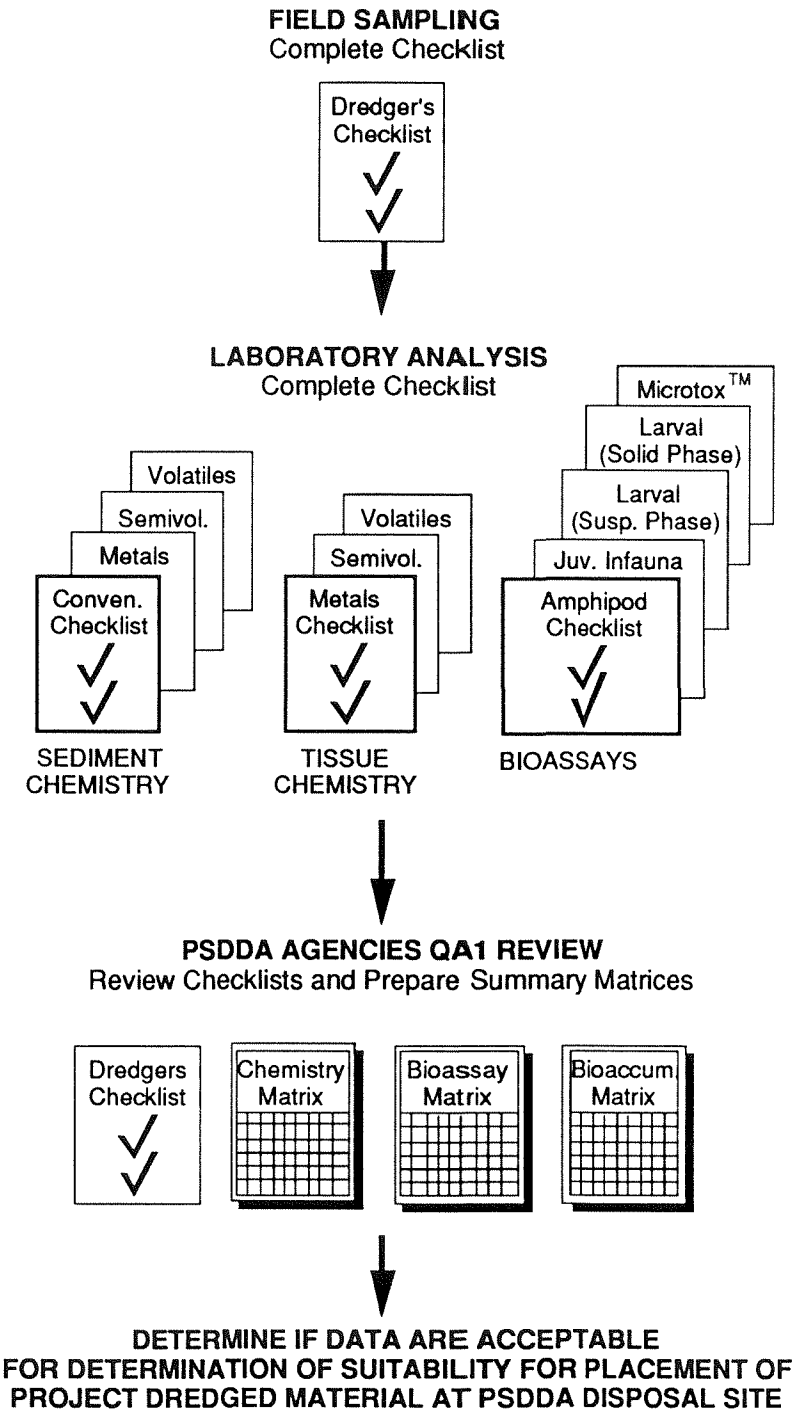

Figure 2. Relationship Between Key Elements of QA1 Data Review and the PSDDA Process

The data package is then sent to the Corps as lead PSDDA agency for a coordinated QA1 review. The Corps assembles the QA1 checklists and enters relevant QA information contained in the checklist into chemistry and bioassay summary matrices. These summary matrices are then used to determine if the data are acceptable for permitting purposes.

1.4. REPORT ORGANIZATION

PSDDA requirements for sampling and analytical strategies and the way in which these requirements are integrated into QA1 are summarized in Section 2. Section 3 describes formats for the QA1 checklists and summary matrices. In Section 4, guidance for determining the acceptability of QA1 data is provided. A glossary of terms used in this document and pertaining to QA in general is included. Definitions were taken from the PSDDA (1988b) and PSEP (1986) protocols. Required laboratory documentation is presented in Appendix A. Example checklists are provided in Appendix B.

2. KEY ELEMENTS OF PSDDA AND THEIR INTEGRATION INTO QA1

In this section, PSDDA sampling strategies and chemical and biological analytical requirements are described. Elements of the PSDDA evaluation procedures that are integrated into QA1 are also described.

2.1. PSDDA SAMPLING STRATEGY

Field sampling strategies for the individual dredging projects will be discussed and refined in pre-application meetings between the dredgers, dredging contractors, and PSDDA agencies. PSDDA sampling strategies are designed to support either the FC or PC of a potential dredging site. FC of dredged material includes sampling, compositing, and testing as a basis for regulatory decisions on the acceptability of the material for unconfined, open-water disposal. For relatively large projects, the dredger may elect to perform PC of sediments contained in the proposed dredging area if the dredger feels the project area is overranked. The major requirements for PSDDA field sampling efforts are described in PSDDA (1988b). Recommended requirements for dredged material disposal assessments were defined by a regional administrative decision (see Exhibit B in PSDDA 1988b).

2.1.1. Number and Location of Sediment Samples

For FC, the number of required samples and analyses is based on the volume of sediment involved, the suspected degree of sediment contamination, and the anticipated dredging cut depth. The suspected degree of chemical contamination is represented by an area ranking, which is determined from available information on chemical concentrations and biological effects and the proximity of the area to known and historical sources. The following steps from PSDDA (1988b) are used to determine the sampling strategy for FC:

1. Determine area ranking
2. Estimate the dredging volume above (i.e., surface) and below (i.e., subsurface) the 4-foot cut line
3. Using the sampling and analytical guidelines for the appropriate ranking (see Table II.4-2 in PSDDA 1988b), calculate the minimum number of samples and analyses required for the project
4. Determine the dredging plan (e.g., dredging cut in lifts, cuts from the base of the slope, or cuts by completing successive segments of the channel)
5. Define dredged material management units based on the calculated number of analyses and the dredging volumes above and below the 4-foot cut lines
6. Allocate the calculated number of samples (i.e., designate location of samples in the dredging units)
7. Determine the sample compositing plan (i.e., which samples to mix together).

Key factors that influence the sampling strategy are the area ranking and the spatial configuration of the required dredged volume. The configuration of the dredging volume is determined by the specific needs of the individual dredging projects. The area ranking, on the other hand, is determined by the location of the dredging project, although ranking can also be conducted within a specific project.

PC is intended to provide a reasonable level of confidence in support of a project reranking decision by the appropriate regulatory agencies (Corps, Ecology, EPA) at a relatively low cost. PC is not a substitute for FC but only a means of establishing a "reason to believe" that a lower level of ranking is appropriate. PC data may be used as FC data only for the upper 4 feet of the dredging prism. PC can be applied to an entire project area or a specified subarea. For the option of lowering the ranking one level, the number of PC samples would be 10 percent of the FC minimum surface sample requirements but never less than two samples per project. For the option of lowering the ranking two levels, the number of PC samples would be 20 percent of the FC minimum surface sample requirements but never less than three samples per project. All samples must be analyzed for all PSDDA chemicals of concern, and no sample compositing is allowed. The area or subarea is then evaluated on a worst-case basis (i.e., the sample having the highest level of a contaminant of concern will be the basis of the ranking). Details of PC are described in PSDDA (1988b).

2.1.2. Sampling and Testing Sequence

The sampling and testing sequence developed by PSDDA is based on a tiered approach to the evaluation of sediment quality. Tier 1 consists of an evaluation of the project area to determine if the sediments may contain chemicals of concern. If sediments are suspected to contain chemicals of concern, chemical testing (Tier 2) is required. When no contamination is expected, chemical characterization of at least one sample is required. Biological testing (Tier 3) is required only if chemical concentrations fall within a certain range; however, dredgers have the option to conduct biological testing even if chemical concentrations fall outside the designated concentration range. Consequently, there are three sampling alternatives for the dredging applicant:

1. Collect sufficient sediment for all chemical and biological tests potentially required and run these tests concurrently.
2. Collect sufficient sediment as above, but archive some material pending the results of the chemical analyses.
3. Collect only enough sediment to conduct the chemical analyses and, if biological testing is required, resample the site.

The sampling and testing sequence may be selected by the dredger through coordination with the PSDDA agencies. Alternative 1 is the least time-consuming and is likely the most cost-effective when the need for biological testing is expected. The use of Alternatives 1 and 2 is encouraged, because they provide chemical and biological information on subsamples of one sediment sample. If Alternative 2 is selected, storage procedures for sediments to be used for biological testing have been recommended (PSDDA 1988b) to allow chemical testing to be completed first. Special time constraints are imposed on the QA1 review of chemical data if Alternative 2 is selected, because the need for biological testing is contingent on the results of chemical data. For Alternative 3, biological testing of sediments resampled at the same stations without reanalysis of the sediment

chemistry is allowed. Time constraints for QA1 review also exist for Alternative 3, because it may be necessary to remobilize field crews and resample the project area to collect biological samples.

2.2. SPECIFICATION OF ANALYTICAL VARIABLES, PROTOCOLS, AND CONTROL LIMITS

Chemical and/or biological testing may be required for sediment designated for dredging. In this section, appropriate variables, protocols, and control limits are described.

2.2.1. Chemical Variables, Protocols, and Control Limits

Only certain chemicals are routinely considered in the evaluation of dredged material. These chemicals of concern were identified by considering toxicological information for chemicals known to be found in Puget Sound. The 58 chemicals required for all surveys, listed in Table 1, have one or more of the following characteristics: a demonstrated or suspected effect on human health, a widespread distribution or high concentration relative to natural conditions, a potential for remaining toxic for a long time in the environment, and a potential to bioaccumulate and enter the food web.

An additional nine chemicals, listed as required for selected surveys in Table 1, will only be measured in certain areas, or for reasons other than concern for biological effects (e.g., as chemical tracers, such as manganese). Other selected chemicals are analyzed for individual projects located near specific sources of chemicals of concern that do not exhibit a widespread distribution. For example, guaiacol, chlorinated guaiacols, butyltins, and chromium were identified by EPWG as important in localized areas. Conventional chemical variables are also measured routinely to further characterize the sediments and to support the interpretation; however, these variables are not considered chemicals of concern.

The digestion technique used to extract metals from sediments has recently undergone review. As a result, PSDDA (1989) recommends the total acid digest (TAD) method, which employs hydrofluoric acid and aqua regia to completely break down the mineral matrix. However, a second technique, the strong acid digest (SAD) is also allowed if control limits can be met. The advantages and disadvantages of the two techniques are summarized in PSDDA (1989).

Chemical sampling and testing protocols to be used with the PSDDA procedures are generally those recommended by PSEP in *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound* (PSEP 1986). Certain elements of the protocols are in the process of being updated or amended. Both PSDDA evaluation procedures and PSEP protocols are subjected to periodic review to ensure that they reflect technical advances and pertinent changes in the regulatory environment. Consequently, this document will evolve to reflect these changes. When available and wherever possible, the PSEP protocols (and revisions) will be required for dredged material sampling and testing. However, there are exceptions to these requirements (see PSDDA 1988b). For example, sediment ammonia analysis is not currently addressed in the PSEP protocols. Ammonia analysis by the standard procedures of EPA/Corps is specified by PSDDA (described in Plumb 1981).

PSDDA chemical detection limits (DL) and SL are summarized in Table 2. PSDDA requires that chemical measurements be quantified at SL levels but recommends that DL be achieved as well. Recent modifications to these DL have been made. For metal samples digested using the

TABLE 1. CHEMICAL VARIABLES

REQUIRED FOR ALL SURVEYS

Conventional Variables

Total volatile solids (TVS)	Grain size distribution	Total sulfides
Total organic carbon (TOC)	Percent solids	Ammonia

Metals and Metalloids

Antimony	Copper	Nickel
Arsenic	Lead	Silver
Cadmium	Mercury	Zinc

Semivolatile Organic Compounds (Acid/Base/Neutral)

Phenols (organic acids)

Phenol	2,4-Dimethylphenol
2-Methylphenol	Pentachlorophenol
4-Methylphenol	

Low Molecular Weight Aromatic Hydrocarbons (neutrals)

Naphthalene	Fluorene
2-Methylnaphthalene	Phenanthrene
Acenaphthylene	Anthracene
Acenaphthene	

High Molecular Weight Aromatic Hydrocarbons (neutrals)

Fluoranthene	Benzo(a)pyrene
Pyrene	Indeno(1,2,3-c,d)pyrene
Benz(a)anthracene	Dibenzo(a,h)anthracene
Chrysene	Benzo(g,h,i)perylene
Benzofluoranthenes	

Chlorinated Aromatic Hydrocarbons (neutrals)

1,2-Dichlorobenzene	1,2,4-Trichlorobenzene
1,3-Dichlorobenzene	Hexachlorobenzene (HCB)
1,4-Dichlorobenzene	

Chlorinated Aliphatic Hydrocarbons (neutrals)

Hexachlorobutadiene	Hexachloroethane

TABLE 1. (Continued)

Semivolatile Organic Compounds (Acid/Base/Neutral) (continued)

Phthalate Esters (neutrals)

Dimethyl phthalate
Diethyl phthalate
Di-n-butyl phthalate
Butyl benzyl phthalate
Bis(2-ethylhexyl)phthalate
Di-n-octyl phthalate

Miscellaneous Oxygenated Compounds

Benzyl alcohol
Dibenzofuran
Benzoic acid

Organonitrogen Compounds (organic bases)

N-nitrosodiphenylamine

PCB

Total PCB

Pesticides

Total DDTs (p,p)
Heptachlor
Alpha-chlordane
Aldrin
Dieldrin
Gamma-HCH (lindane)

Volatile Organic Compounds

Trichloroethene
Tetrachloroethene
Ethylbenzene
Total xylenes

REQUIRED FOR SELECTED SURVEYS

Manganese
Chromium
Trichlorobutadiene isomers
Tetrachlorobutadiene isomers
Pentachlorobutadiene isomers
Tetrabutyltin
Tributyltin
Dibutyltin
Monobutyltin
2-Methoxyphenol (guaiacol)
3,4,5-Trichloroguaiacol
4,5,6-Trichloroguaiacol
Tetrachloroguaiacol

TABLE 2. RECOMMENDED CHEMICAL DETECTION LIMITS FOR SEDIMENT AND TISSUE MATRICES AND PSDDA SCREENING LEVELS

Chemical	Sediment (dry weight) PSDDA SL[a]	Sediment (dry weight) PSDDA Detection Limit[b]	Tissue (wet weight) PSDDA Detection Limit[b]
Metals and Metalloids (mg/kg; ppm)			
Antimony	2.6	0.1	0.02
Arsenic	70	0.1	0.02
Cadmium	0.96	0.1	0.01
Copper	81	0.1	0.01
Lead	66	0.1	0.03
Mercury	0.21	0.01	0.01
Nickel	140 (28)[c]	0.1	0.02
Silver	1.2	0.1	0.01
Zinc	160	0.2	0.2
Organic Compounds (μg/kg; ppb)			
Low molecular weight PAH	610	1-50	10-20
Naphthalene	210	1-50	10-20
Acenaphthylene	64	1-50	10-20
Acenaphthene	63	1-50	10-20
Fluorene	64	1-50	10-20
Phenanthrene	320	1-50	10-20
Anthracene	130	1-50	10-20
2-Methylnaphthalene	67	1-50	10-20
High molecular weight PAH	1,800	1-50	10-20
Fluoranthene	630	1-50	10-20
Pyrene	430	1-50	10-20
Benz(a)anthracene	450	1-50	10-20
Chrysene	670	1-50	10-20
Benzofluoranthenes	800	1-50	10-20
Benzo(a)pyrene	680	1-50	10-20
Indeno(1,2,3-c,d)pyrene	69	1-50	10-20
Dibenzo(a,h)anthracene	120	1-50	10-20
Benzo(g,h,i)perylene	540	1-50	10-20
Chlorinated benzenes			
1,3-Dichlorobenzene	170	1-50	10-20
1,4-Dichlorobenzene	26	1-50	10-20
1,2-Dichlorobenzene	19	1-50	10-20
1,2,4-Trichlorobenzene	6.4	1-50	10-20
Hexachlorobenzene	23	1-50	10-20

TABLE 2. (Continued)

Chemical	Sediment (dry weight) PSDDA SL[a]	Sediment (dry weight) PSDDA Detection Limit[b]	Tissue (wet weight) PSDDA Detection Limit[b]
Organic Compounds (μg/kg; ppb) (continued)			
Total PCB	130	1-50	10-20
Phthalates			
Dimethyl phthalate	160	1-50	10-20
Diethyl phthalate	97	1-50	10-20
Di-n-butyl phthalate	1,400	1-50	10-20
Butyl benzyl phthalate	470	1-50	10-20
Bis(2-ethylhexyl)phthalate	3,100	1-50	10-20
Di-n-octyl phthalate	6,200 (69,000)[c]	1-50	10-20
Phenols			
Phenol	120	1-50	10-20
2-Methylphenol	10	1-50	10-20
4-Methylphenol	120	1-50	10-20
2,4-Dimethylphenol	10	1-50	10-20
Pentachlorophenol	69 (140)[c]	1-50	10-20
Miscellaneous extractable compounds			
Benzyl alcohol	10	1-50	10-20
Benzoic acid	216	1-50	10-20
Dibenzofuran	54	1-50	10-20
Hexachloroethane	1,400	1-50	10-20
Hexachlorobutadiene	29	1-50	10-20
N-nitrosodiphenylamine	22	1-50	10-20
Volatile organic compounds			
Trichloroethene	160	10-20	5-10
Tetrachloroethene	14	10-20	5-10
Ethylbenzene	10	10-20	5-10
Total xylenes	12	10-20	5-10
Pesticides			
Total DDT	6.9	1-50	10-20
Aldrin	5	1-50	10-20
Chlordane	5	1-50	10-20
Dieldrin	5	1-50	10-20
Heptachlor	5	1-50	10-20
Lindane	5	1-50	10-20

[a] PSDDA screening level (PSDDA 1989).

[b] Recommended range cited in PSEP (1986).

[c] PSDDA screening level (PSDDA 1988b).

TAD method, it has been recognized that these DL may not always be achievable because of matrix interference problems and method-imposed sample size limitations (PSDDA 1989). Because DL for many metals were well below SL, the following recommendation were made:

- Extracted sample size would be increased from 0.2 to approximately 0.3 grams to provide a stronger signal
- Certified reference material (CRM) or standard reference material (SRM) should be run using the matrix matching technique for quality control
- The associated DL for this technique must fall within a factor of 2 of the DL cited in Table 2.

QA1 review entails the comparison of DL to SL, but QA2 review involves comparisons to recommended DL (the former comparison focuses on regulatory concerns, whereas the latter focuses on analytical concerns). Required frequencies and control limits for chemistry QA samples are summarized in Table 3.

2.2.2. Biological Variables, Protocols, and Control Limits

Biological tests can be conducted at the dredger's option but are required if the concentrations of chemicals of concern fall within a specified range. When biological testing is indicated, the following acute biological tests may be used:

- **Amphipod mortality bioassay (10-day exposure)**—The amphipod *Rhepoxynius abronius* is exposed to test sediments for 10 days, after which the surviving individuals are counted.
- **Juvenile infauna mortality bioassay (10-day exposure)**—Juveniles of an infaunal species are exposed to test sediments for 10 days, after which the surviving individuals are counted. The test can be conducted using the polychaete *Neanthes arenaceodentata*, the Pacific oyster (*Crassostrea gigas*), or the native littleneck clam (*Protothaca staminea*).
- **Sediment larval bioassay (solid and suspended phases)**—During the early stages of embryonic development, fertilized eggs of a bivalve or echinoderm species normally develop into free-swimming larvae. In the presence of test sediments (solid phase) or sediment elutriate (suspended phase), larval mortality (i.e., lethal effects) and the proportion of larvae developing abnormally (i.e., sublethal effects) are used as toxicity indicators. The species normally used in this test include the Pacific oyster (*Crassostrea gigas*), the edible mussel (*Mytilus edulis*), the sand dollar (*Dendraster excentricus*), and the purple urchin (*Strongy locentrotus purpuratus*). Exposure periods may differ among species and are currently being defined by PSDDA. Initially, the exposure period for each test will be based on adequate development in the negative controls.
- **Microtox™ bioassay (15-minute exposure)**—The luminescence of the bacterium *Photobacterium phosphoreum* is a product of its electron transport system and thus directly reflects the metabolic state of the cell. Decreased luminescence following exposure to a saline extract of the test sediment provides a quantitative measure of toxicity.

TABLE 3. REQUIRED FREQUENCIES AND CONTROL LIMITS FOR CHEMISTRY QA SAMPLES

Analysis Type	Frequency of Analysis[a]	Control Limit
Organic Compounds		
Method blank	One per extraction batch (semivolatile organics) One per extraction or one per 12-hour shift, whichever is most frequent (volatile organics)	**Phthalate:** 5 μg total or 50% of the analyte **Other organic compounds:** 2.5 μg total or 5% of the analyte
Certified reference material (CRM)	<50 samples: one per set of samples submitted to laboratory >50 samples: one per 50 samples analyzed	95% confidence interval for certified reference material
Matrix spikes	**Not** required if complete isotope dilution used <20 samples: one per set of samples submitted to laboratory ≥20 samples: 5% of total number of samples	50% recovery
Analytical replicates	<20 samples: one per set of samples submitted to laboratory ≥20 samples: one triplicate and additional duplicates for a minimum of 5% total replication	±100% coefficient of variation
Metals/Metalloids[b]		
Preparation blank	5% or one per batch, whichever is more frequent	≤ detection limit
Certified reference material	5% or one per batch, whichever is more frequent	80-120 percent recovery
Matrix spike	5% or one per batch, whichever is more frequent	75-125 percent recovery
Analytical replicates	5% or one per batch, whichever is more frequent	±20 percent RPD or CV

[a] Frequencies listed are minimums; some programs may require higher levels of effort.

[b] For batches of five samples or less, the minimum QC checks should be a preparation blank and the analysis of a CRM. If an analyte is not in the CRM, a matrix spike must be analyzed for that particular analyte. In general, for small batches (i.e., ≤5 samples), the priority of QC checks should be: CRM > analytical duplicates > matrix spikes. If several small batches of the same matrix are analyzed sequentially (i.e., for several small projects), an CRM can be analyzed at a frequency of 5 percent overall, with at least one sample duplicate analyzed per individual batch.

Bioaccumulation tests are required only when chemical concentrations exceed a certain "trigger" level (summarized in Table II-6.2 of PSDDA 1988b). EPWG has chosen to use bioaccumulation data as an indicator of human health effect only. The bioaccumulation test recommended by PSDDA requires a chemical tissue analysis using a bivalve species of the genus *Macoma*. The *Macoma* spp. bioaccumulation test measures tissue residue toxicant in the bivalve over a 30-day period.

Biological sampling and testing protocols to be used with the PSDDA procedures are generally those recommended by PSEP (1986). However, many of the tests required by the PSDDA evaluation are not included in the PSEP protocols. *Macoma* bioaccumulation exposures, juvenile infauna mortality, and sediment larval (suspended-phase) tests are not currently addressed in the PSEP protocols. The following modifications and alternative testing procedures are designated for use in the PSDDA evaluation procedures:

- **Juvenile infauna bioassay**—Standardized protocols are not yet available. PSDDA recommends that the test be run by adapting and utilizing available method guidance (e.g., U.S. EPA and Corps 1977).
- **Sediment larval bioassay**—PSDDA requires that dissolved oxygen in the test chamber be maintained above 4 ppm. For the suspended-phase test, PSDDA specifies that testing be conducted according to U.S. EPA and Corps (1977).
- **Microtox™**—PSDDA requires that the saline extract method be used rather than the organic extract method, which is listed as an option in the PSEP protocols.

PSDDA also specifies that sediments for all bioassays be held ≤6 weeks at 4°C under nitrogen, compared with the PSEP recommendation of 14 days. Biological control limits for holding conditions and times, replicates, negative controls, and experimental conditions are summarized in Table 4.

2.3. SAMPLING AND ANALYTICAL INFORMATION INCLUDED IN QA1

The process of QA1 consists of the systematic distillation and review of information. The relationship between key elements of QA1 data review is illustrated in Figure 2.

The chemistry data set consists of discrete chemical categories and the bioassay data consist of four individual bioassays. Individual checklists are required for each of the following data types:

- Chemistry
  - Conventional parameters
  - Metals
  - Semivolatile organic compounds
  - Volatile organic compounds
- Bioassays
  - Amphipod mortality
  - Juvenile infauna mortality
  - Bivalve larval test (solid phase)

TABLE 4. BIOASSAY CONTROL LIMITS

Bioassay	Sediment Holding Prior to Testing: Conditions	Sediment Holding Prior to Testing: Time	Replicates	Negative Control	Reference Performance[a]	Experimental Conditions
Amphipod mortality	4° C in N atmosphere	≤6 weeks	5	≤10% mortality	≤20% mortality	Temperature = 15±1° lC Salinity = 28±1 ppt pH = 8±1 DO = >5 mg/L
Juvenile infauna mortality	4° C in N atmosphere	≤6 weeks	5	≤10% mortality	<20% mortality	(not established)
Sediment larval test (solid and suspended *Crassotrea gigas* phase)	4° C in N atmosphere	≤6 weeks	5	≤30% mortality ≤10% abnormality	≤20% abnormality	Temperature = 20±1° C Salinity = 28±1 ppt pH = 8±1 DO = >4 mg/L
Mytilus edulis	4° C in N atmosphere	≤6 weeks	5	≤30% mortality ≤10% abnormality	≤20% abnormality	Temperature = 13±1° C Salinity = 28±1 ppt pH = 8±1 DO = >4 mg/L
Dendraster excentricus	4° C in N atmosphere	≤6 weeks	5	≤30% mortality ≤10% abnormality	≤20% abnormality	Temperature = 9±2° C Salinity = 30±3 ppt pH = 8±1 DO = >4 mg/L
Strongylocentrotus purpuratus	4° C in N atmosphere	≤6 weeks	5	≤30% mortality ≤10% abnormality	≤20% abnormality	Temperature = 15±2° C Salinity = 30±3 ppt pH = 8±1 DO = >4 mg/L
Microtox (saline extract)	4° C in N atmosphere	≤6 weeks	2 per dilution	Blank ratios differ by <0.02	none	Temperature = 15±1° C

[a] Performance is expressed as percent above control value.

- Bivalve larval test (suspended phase)
- Microtox™ (saline extract)

- Bioaccumulation
  - Metals
  - Semivolatile organic compounds
  - Volatile organic compounds.

The formats for the sediment chemistry and tissue bioaccumulation checklists are identical.

Key information on sampling and analytical strategies is summarized in the dredger's checklist (Figure 3). This information sets the stage for data review by providing background for the sampling and analytical strategies. The area ranking and the spatial configuration of the sediments to be dredged determine the number of samples required by PSDDA and influence the analytical strategy and sequence, unless PC is undertaken (PSDDA 1988b). Only limited sampling and chemical analyses are required for PC, which provide a basis for reranking a project and thus may determine the number of samples and composites to be done in the subsequent FC.

Key information on the quality of analytical data is summarized in a series of checklists by the laboratories responsible for generating the data (Figure 3). This information includes data completeness, holding conditions, format (i.e., units, significant figures), DL, and the need for the following QA samples:

- **Method blanks**—Method blanks determine the laboratory contributions to analyte concentrations
- **Certified reference materials (CRM)**—CRM are used to assess the accuracy of an analysis and to compare performance among laboratories. CRM should be of a matrix similar to the samples collected (e.g., sediment, fish tissue) and contain, if possible, the major chemicals of concern at concentrations near those in the samples. CRM containing all chemicals of concern are not generally available. However, uncertified sediment reference materials containing many of the chemicals of concern are available in the Puget Sound area. Reference materials that have not been certified are of less value in assessing accuracy than CRM.
- **Replicates**—Laboratory replicate analyses provide information on the precision (repeatability) of analytical results. Triplicates yield a more meaningful statistical measure of variability than duplicates.
- **Matrix spikes**—Matrix spikes, like CRM, are used to evaluate accuracy. Matrix spikes offer the advantage of being site-specific to the area tested and can include all the chemicals of concern; however, matrix spikes are not necessarily representative of the extraction efficiency that would be attained for environmentally contaminated samples.

Key bioassay information (Figure 3) includes data completeness, holding conditions and times, format (i.e., percent mortality and water quality variables for each replicate), and the need for the following QA elements:

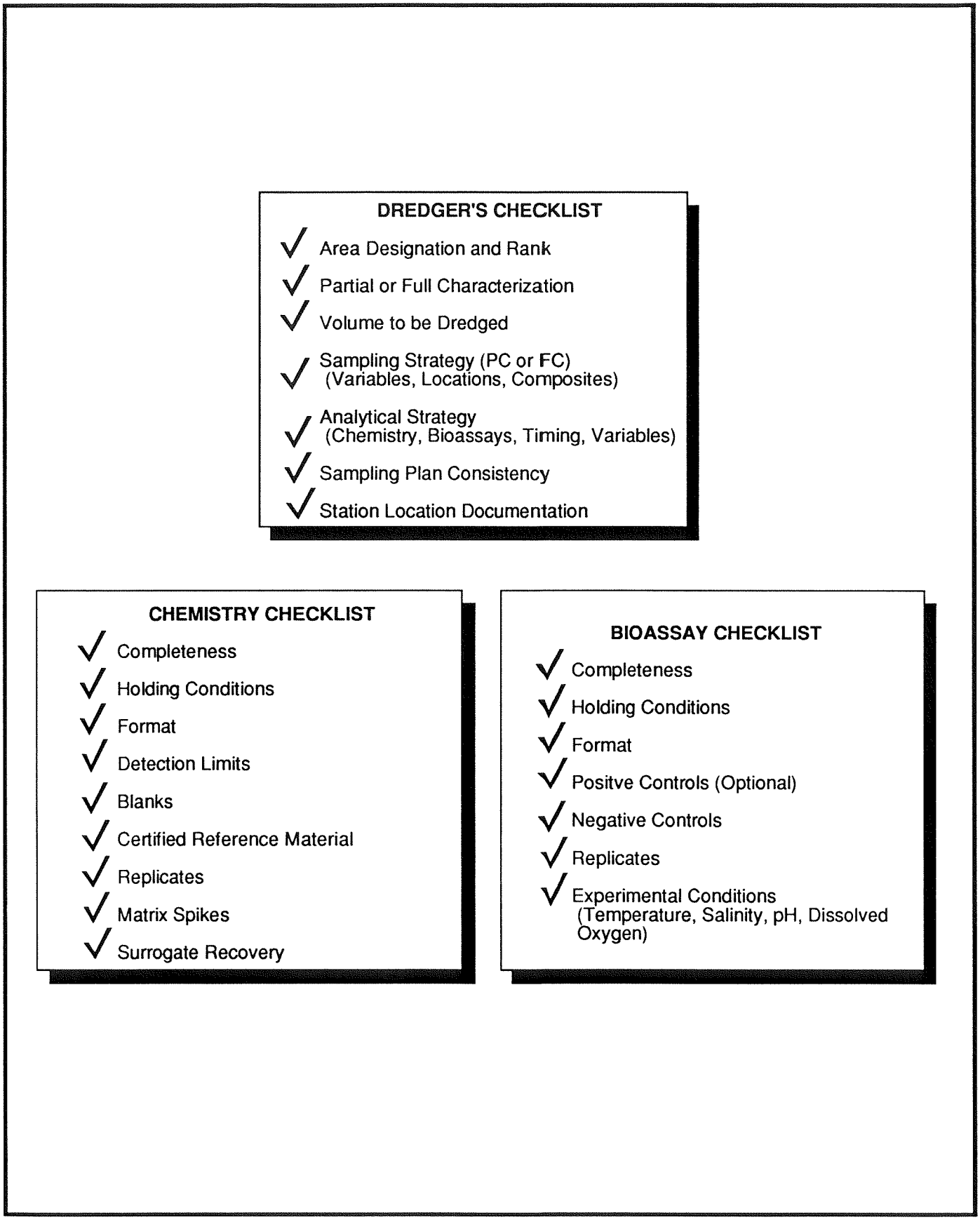

Figure 3. Information Included in QA1 Checklists

- **Positive controls**—Positive controls are tests conducted using a reference toxicant to ensure test organisms are suitably responsive to toxic effects. Although not required by PSDDA, positive controls are recommended by PSEP.
- **Negative controls**—Negative controls are tests conducted using clean (preferably native) water and/or sediments to ensure test organisms are suitably healthy for testing.
- **Replicates**—Replicates are repeated analyses on subsamples of the same field sample to improve the estimate of mean bioassay response and to assess response variability.
- **Experimental conditions**—Experimental conditions are the physical/chemical conditions (e.g., temperature, salinity, dissolved oxygen) under which each bioassay is conducted. These conditions must be standardized to ensure a differential bioassay response does not manifest among different studies.

In addition to the above QA elements, PSDDA requires that reference sediments be analyzed. Results are then compared with bioassay responses to test sediments to determine whether the latter responses are significant. Reference samples should be relatively uncontaminated and should have conventional characteristics (e.g., grain size) similar to the test samples. In this manner, observed differences in bioassay responses between reference and test sediments can be attributed with a relatively high degree of confidence to chemical toxicity rather than to conventional variables. Detailed specifications of QA elements and reference area performance standards for each bioassay are presented in Table 4.

Checklists are reviewed by agency personnel, and key information (i.e., cases where data quality objectives have not been met) is entered into chemistry and bioassay summary matrices. The summary matrices support the interpretation of QA l results and provide the decision-base for determining if the data are acceptable for issuing dredging permits.

3. QA1 CHECKLIST FORMATS

In this section, the format for field and laboratory checklists is described. These checklists are filled out by field teams and qualified laboratory personnel, respectively. Additional summary information can be added to the chemistry checklists by PSDDA data reviewers if the appropriate computerized data are available. At the conclusion of this section, the format for the summary matrix is presented. The summary matrix, prepared by PSDDA agency personnel, contains the key QA1 information needed to determine if the data are acceptable for permitting. Example checklist and summary matrices are provided in Appendix B.

3.1. DREDGERS CHECKLIST

The dredger's checklist is presented in Table 5. The sampling and analytical strategies summarized in the dredger's checklist are determined from the project area ranking (Phase I area rankings are defined in PSDDA 1988b) and the pre-application meetings between the dredgers, dredging contractors, and PSDDA agencies. Portions of the dredger's checklist (i.e., variables sampled and number of samples) must be filled out during or immediately after field sampling to document compliance with the sampling and analytical strategies determined in the pre-application meetings.

3.2. CHECKLIST FOR CONVENTIONAL VARIABLES IN SEDIMENT

The conventional variables checklist is presented in Table 6. QA1 measures for sediment conventional variables are less extensive than those for chemical and biological variables. PSEP specifies frequencies of QA analyses for sediment conventional variables (e.g., how often to analyze analytical replicates) but does not specify control limits for data review. Nonetheless, blanks, replicates, and reference materials are recommended for at least some sediment conventional variables (e.g., TOC analysis) and are included in the checklist.

3.3. CHECKLIST FOR METALS IN SEDIMENT

The metals checklist is presented in Table 7. QA1 measures for metals analyses include preparation blanks, CRM, analytical replicates, and matrix spikes. Additional summary information regarding DL and relative blank contamination can be added by PSDDA data reviewers if the appropriate computerized data are available (see end of Table 7).

3.4. CHECKLIST FOR SEMIVOLATILE ORGANIC COMPOUNDS (A/B/N AND PCB/PESTICIDES) IN SEDIMENT

The semivolatile organic compounds checklist is presented in Table 8. QA1 measures for semivolatile organic compounds include method blanks, certified reference materials, analytical replicates, and matrix spikes. Additional summary information regarding DL, surrogate recoveries,

[PSDDA Tracking Number]

TABLE 5. DREDGER'S CHECKLIST

Project Name _____ Applicant _____

Location _____ Date _____

Area Rank(s) _____

Partial characterization (PC) or full characterization (FC)? _____

Total volume of sediment to be dredged (attach diagram) _____

SAMPLES COLLECTED

	Chemistry	Bioassays	Bioaccumulation
# Stations			
# Field samples[a]			
# Study samples[b]			
Shipboard storage conditions			

ANALYTICAL STRATEGY

Chemistry only _____ Synoptic chemistry and biology _____

Biology only _____ Chemistry 1st, biology 2nd _____

TARGET VARIABLES

	Chemistry	Bioaccumulation
All PSSDA chemicals of concern		
Additional chemicals		
Deleted chemicals		

FIELD COLLECTION

What positioning method was used? (describe) _____

Station positioning information attached? _____

Sampling strategy (attach diagram) _____

Sampling device _____

Were chemistry/bioassay samples homogenized prior to subsampling? _____

Were corresponding chemistry and bioassay subsamples taken from the same composite? _____

[a] Field samples includes all samples collected from discrete coordinates in a dredging volume.
[b] Study samples includes all samples remaining after compositing has been conducted.

TABLE 6. CHECKLIST FOR CONVENTIONAL VARIABLES IN SEDIMENT

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date: Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

COMPLETENESS AND HOLDING CONDITIONS

	TOC	TVS	Total Sulfides	Ammonia	Total Solids	Grain Size Distribution
Method (identify)	_____	_____	_____	_____	_____	_____
# Samples submitted	_____	_____	_____	_____	_____	_____
# Samples analyzed	_____	_____	_____	_____	_____	_____

Holding conditions acceptable? (Y/N) _____

(see final page of checklist for guidelines)

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Sample detection limits provided, when applicable (total sulfides, ammonia) _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

_____

TABLE 6. (Continued)

QA/QC SAMPLES

Method Blank

TOC Total # _____

Frequency _____
(minimum 1 per 20 samples)[a]

Amount detected in blank _____
(no PSEP control limit)

Certified Reference Materials

TOC Total # _____

Frequency _____
(minimum 1 per survey)

CRM used _____

Within 95% confidence interval? _____
(not a PSEP control limit)

Analytical Replicates

	TOC	TVS	Total Sulfides	Ammonia	Total Solids	Grain Size Distribution
Total #	_____	_____	_____	_____	_____	_____
Frequency (minimum 1 triplicate per 20 samples)[a]	_____	_____	_____	_____	_____	_____

[a] Recommended by PSEP (1986).

TABLE 6. (Continued)

RECOMMENDED SAMPLE SIZES, CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR SEDIMENT CONVENTIONAL VARIABLES (PSEP 1986)

Variable	Minimum Sample Size (grams)[a]	Container[b]	Preservation	Maximum Holding Time
Particle size	100-150[c]	P,G	Cool, 4° C	6 months[d]
Total solids	50	P,G	Freeze	6 months[d]
Total volatile solids	50	P,G	Freeze	6 months[d]
Total organic carbon	25	P,G	Freeze	6 months[d]
Total sulfides	50	P,G	Cool, 4° C, 1N zinc acetate	7 days[d]
Ammonia	20	P,G	Cool, 4° C (minimize air contact, keep field moist)	7 days[e]

[a] Recommended field sample sizes for one laboratory analysis. If additional laboratory analyses are required (e.g., replicates), the field sample size should be adjusted accordingly.

[b] P = polyethylene; G = glass.

[c] Sandy sediments require larger sample sizes than do muddy sediments.

[d] This is a suggested holding time. No EPA criteria exist for the preservation of this variable.

[e] This holding time is recommended by Plumb (1981).

TABLE 7. CHECKLIST FOR METALS IN SEDIMENT

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted[a]? (Y/N)_____

Digestion procedure [Total Acid Digest (TAD) or Strong Acid Digest (SAD)] _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) [6 months frozen for metals except mercury; 28 days frozen (in glass) for mercury] _____

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

Sample detection limits (DL) provided for each analyte? (Y/N) _____

_____

TABLE 7. (Continued)

QA/QC SAMPLES

Preparation Blank

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals observed above detection limits in one or more blanks[c]

_____ _____

_____ _____

Certified Reference Materials

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

CRM used _____

Chemicals outside 80-120% recovery[c]

_____ _____

_____ _____

(for chemicals without certified values, use matrix spike results)

Analytical Replicates

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Samples/chemicals with >20% relative percent difference (RPD) or coefficient of variation (CV)[c]

_____ _____

_____ _____

Matrix Spikes

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals with recovery outside 75-125%[c]

_____ _____

_____ _____

TABLE 7. **(Continued)**

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

Antimony _____ Arsenic _____ Cadmium _____

Copper _____ Lead _____ Mercury _____

Nickel _____ Silver _____ Zinc _____

(see Table 2)

Preparation Blanks (Relative blank contamination)

Are sample results <5 times blank values in any samples? (Y/N) _____

If yes, identify elements and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤5 samples should be as follows: CRM > analytical replicate > matrix spikes.

[c] PSEP control limit.

[PSDDA Tracking Number]

TABLE 8. CHECKLIST FOR SEMIVOLATILE ORGANIC COMPOUNDS IN SEDIMENT

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted[a]? (Y/N) _____

Analytical method _____

COMPLETENESS AND HOLDING CONDITIONS

	# Samples Submitted	# Samples Analyzed
A/B/N	_____	_____
Pesticides/PCB	_____	_____

Holding conditions acceptable? (Y/N) (1 year for frozen sediment)

_____

If no, identify samples _____

Extract holding times acceptable? (Y/N) _____

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

_____

TABLE 8. (Continued)

Sample detection limits (DL) provided for each analyte? (Y/N) _____

QA/QC SAMPLES

Method Blank

Total # _____

Frequency _____
(minimum 1 per extraction batch)[b]

Chemicals detected above 5 μg total (for phthalates) and 2.5 μg total[b]
(for other organic compounds; lower levels may be appropriate for pesticides and PCBs)

_____ _____

_____ _____

Certified Reference Materials

Total # _____

Frequency _____
(<50 samples - 1 per set of samples submitted to lab; >50 samples - 1 per 50 samples analyzed)[b]

CRM used _____

Chemicals outside 95% confidence interval (for certified values)[b]

_____ _____

_____ _____

Analytical Replicates

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicate for minimum of 5% total replication)[b]

Samples/chemicals with >100% RPD or CV[b]

_____ _____

_____ _____

Matrix Spikes (not required for A/B/N if isotope dilution used)

Total # _____

Frequency[c] _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 5% of total samples)[b]

Chemicals with <50% recovery[b]

_____ _____

_____ _____

TABLE 8. (Continued)

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

A/B/N (PAH) _____ A/B/N (phenols, benzoic acid, benzyl alcohol) _____ A/B/N (other) _____

PCB _____ Pesticides _____

(see Table 2)

Surrogate Recovery (A/B/N)

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10 percent recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50 percent recovery[b] (also identify samples)

_____ _____

_____ _____

Surrogate Recovery (Pesticides/PCBs)

Were surrogates added to all samples?[b] (Y/N) _____

Identify samples with <50 percent surrogate recovery[b]

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

For target compounds other than phthalates, was blank contamination >5 percent of any sample concentrations?[b] (Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

TABLE 8. (Continued)

For phthalates, was blank contamination >50 percent of any sample concentration?[b]

(Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤ 5 samples should be as follows: CRM > analytical replicate > matrix spikes.

[c] PSEP control limit.

and relative blank contamination can be added by PSDDA data reviewers if the appropriate computerized data are available (see end of Table 8). Acid/base/neutral (A/B/N) compounds and PCB/pesticides have been combined in this checklist to facilitate QA1 review, although QA2 review would require that these compound classes be treated separately because they represent discrete instrumental analyses. A/B/N and PCB/pesticides are typically analyzed by different instrumental methods [gas chromatography/mass spectrophotometry (GC/MS) and gas chromatography/electron capture detection (GC/ECD), respectively] using subsamples from the same extract. It is intended that the control limits in this checklist refer to analyses of A/B/N compounds and PCB/pesticides.

3.5. CHECKLIST FOR VOLATILE ORGANIC COMPOUNDS IN SEDIMENT

The volatile organic compounds checklist is presented in Table 9. QA1 measures for volatile organic compounds are slightly different than those for semivolatile organic compounds, largely because CRM are not available for volatile organic compounds. The checklists for semivolatile organic compounds and volatile organic compounds have been separated, because the laboratory procedures for the two compound classes are completely different (with "extraction" by gas purging for volatile compounds vs. solvent extraction for semivolatile compounds) and are conducted on different subsamples. Of the chemicals of concern, volatile organic compounds are generally the most likely to be affected by extended holding times.

3.6. CHECKLISTS FOR BIOACCUMULATION DATA

Bioaccumulation checklists (Tables 10-12) have the same format as the sediment chemistry checklists. Depending on the nature of specific projects, certain chemicals may be omitted from bioaccumulation analysis (e.g., volatile organic compounds). Although not included in the checklist for semivolatile organic compounds in tissue, tissue samples may include analyses of lipids (total extractable organic matter).

3.7. CHECKLIST FOR AMPHIPOD MORTALITY BIOASSAY

The amphipod mortality bioassay checklist is presented in Table 13. QA1 measures for the amphipod mortality bioassay include evaluation of interstitial salinities (i.e., in the field), holding conditions, holding times, positive and negative controls, response variability, and experimental conditions.

3.8. CHECKLIST FOR JUVENILE INFAUNA BIOASSAY

The juvenile infauna bioassay checklist is presented in Table 14. QA1 measures for the juvenile infauna bioassay include evaluation of holding times, and positive and negative controls.

3.9. CHECKLISTS FOR SEDIMENT LARVAL BIOASSAY

The sediment larval abnormality (solid phase and suspended phase) bioassay checklists are presented in Tables 15 and 16, respectively. QA1 measures for the bivalve larval abnormality bioassay include evaluation of holding conditions, holding times, positive and negative controls, and experimental conditions.

TABLE 9. CHECKLIST FOR VOLATILE ORGANIC COMPOUNDS IN SEDIMENT

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N) _____

Analytical method _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (14 days at 4° C) _____

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

Sample detection limits (DL) provided for each analyte? (Y/N) _____

_____

TABLE 9. (Continued)

QA/QC SAMPLES

Method Blank

Total # _____

Frequency _____
(minimum 1 per batch or 1 per 12-hour shift, whichever is more frequent)[b]

Chemicals detected above 2.5 μg total in one or more blanks[b]

_____ _____

_____ _____

Analytical Replicates

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicates for 5% replication overall)[b]

Samples/chemicals with >100% RPD or CV[b]

_____ _____

_____ _____

Matrix Spikes (not required if isotope dilution used)

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted; ≥20 samples - 5% overall)[b]

Chemicals with <50% recovery[b]

_____ _____

_____ _____

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

Volatiles _____

(see Table 2)

TABLE 9. (Continued)

Surrogate Recovery

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10 percent recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50 percent recovery[b]

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

Was blank contamination >5 percent of any sample concentrations?[b] (Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] PSEP control limit.

TABLE 10. CHECKLIST FOR METALS IN TISSUE

Project Name _____ Laboratory Report _____

Type of Tissue _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N)_____

Digestion procedure _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (6 months frozen for metals except mercury; 28 days frozen for mercury) _____

If no, identify samples_____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

Sample detection limits (DL) provided for each analyte? (Y/N) _____

_____

TABLE 10. (Continued)

QA/QC SAMPLES

Preparation Blank

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals observed above detection limits in one or more blanks[c]

_____ _____

_____ _____

Certified Reference Materials

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

CRM used _____

Chemicals outside 80-120% recovery[c]

_____ _____

_____ _____

(for chemicals without certified values, use matrix spike results)

Analytical Replicates

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Samples/chemicals with >20% relative percent difference (RPD) or coefficient of variation (CV)[c]

_____ _____

_____ _____

Matrix Spikes

Total # _____

Frequency[b] _____
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals with recovery outside 75-125%[c]

_____ _____

_____ _____

TABLE 10. (Continued)

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed PSSDA recommended limits? (Y/N) _____

If yes, detection limits exceeding PSSDA recommended limits (identify samples)

Antimony _____	Arsenic _____	Cadmium _____
Copper _____	Lead _____	Mercury _____
Nickel _____	Silver _____	Zinc _____

(see Table 2 and supporting text)

Preparation Blanks (Relative blank contamination)

Are sample results <5 times blank values in any samples? (Y/N) _____

If yes, identify elements and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤5 samples should be as follows: CRM > analytical replicate > matrix spikes.

[c] PSEP control limit.

TABLE 11. CHECKLIST FOR SEMIVOLATILE ORGANIC COMPOUNDS IN TISSUE

Project Name _____ Laboratory Report _____

Type of Tissue _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N) _____

Analytical procedure _____
(including extraction and cleanup)

COMPLETENESS AND HOLDING CONDITIONS

	# Samples Submitted	# Samples Analyzed
A/B/N	_____	_____
Pesticides/PCB	_____	_____

Holding conditions acceptable? (Y/N) (6 months for frozen tissue) _____

If no, identify samples _____

Extract holding times acceptable? (Y/N) _____

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected) _____

_____

_____

_____

TABLE 11. (Continued)

Sample detection limits (DL) provided for each analyte? (Y/N) _____

QA/QC SAMPLES

Method Blank

Total # _____

Frequency _____
(minimum 1 per extraction batch)[b]

Chemicals detected above 5 μg total (for phthalates) and 2.5 μg total[b]
(for other organic compounds; lower levels may be appropriate for pesticides and PCBs)

_____ _____

_____ _____

Certified Reference Materials

Total # _____

Frequency _____
(<50 samples - 1 per set of samples submitted to lab; >50 samples - 1 per 50 samples analyzed)[b]

CRM used _____

Chemicals outside 95% confidence interval (for certified values)[b]

_____ _____

_____ _____

Analytical Replicates

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicate for minimum of 5% total replication)[b]

Samples/chemicals with >100% RPD or CV[b]

_____ _____

_____ _____

Matrix Spikes (not required for A/B/N if isotope dilution used)

Total # _____

Frequency[c] _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 5% of total samples)[b]

Chemicals with <50% recovery[b]

_____ _____

_____ _____

TABLE 11. (Continued)

NOTE: The following informatin will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed PSDDA recommended limits? (Y/N) _____

If yes, detection limits exceeding PSDDA recommended limits[b] (identify samples)

A/B/N (PAH) _____ A/B/N (phenols, benzoic acid, benzyl alcohol) _____ A/B/N (other) _____

PCB _____ Pesticides _____

(see Table 2)

Surrogate Recovery (A/B/N)

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10 percent recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50 percent recovery[b] (also identify samples)

_____ _____

_____ _____

Surrogate Recovery (pesticides/PCBs)

Were surrogates added to all samples?[b] (Y/N) _____

Identify samples with <50 percent surrogate recovery[b]

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

For target compounds other than phthalates, was blank contamination >5 percent of any sample concentration?[b] _____

If yes, identify compounds and samples

_____ _____

_____ _____

TABLE 11. (Continued)

For phthalates, was blank contamination >50 percent of any sample concentration?[b]
(Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] PSEP control limit.

[c] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤5 samples should be as follows: CRM > analytical replicate > matrix spikes.

TABLE 12. CHECKLIST FOR VOLATILE ORGANIC COMPOUNDS IN TISSUE

Project Name _____ Laboratory Report _____

Type of Tissue _____

Lab _____ Lab # _____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N) _____

Analytical procedure (including method of maceration) _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (18 days at 4° C) _____

If no, identify samples _____

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures _____

Qualifiers defined (e.g., U = undetected)

_____

_____

_____

Sample detection limits (DL) provided for each analyte? (Y/N) _____

_____

TABLE 12. (Continued)

QA/QC SAMPLES

Method Blank

Total # _____

Frequency _____
(minimum 1 per batch or 1 per 12-hour shift, whichever is more frequent)[b]

Chemicals detected above 2.5 μg total in one or more blanks[b]

_____ _____

_____ _____

Analytical Replicates

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicates for 5% replication overall)[b]

Samples/chemicals with >100% RPD or c.v.[b]

_____ _____

_____ _____

Matrix Spikes (not required if isotope dilution used)

Total # _____

Frequency _____
(<20 samples - 1 per set of samples submitted; ≥20 samples - 5% overall)[b]

Chemicals with <50% recovery[b]

_____ _____

_____ _____

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed control limits? (Y/N) _____

If yes, detection limits exceeding PSDDA recommended limits (identify samples)

Volatiles _____

(see Table 2)

TABLE 12. (Continued)

Surrogate Recovery

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10% recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50% recovery[b] (also identify samples)

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

Was blank contamination >5 percent of any sample concentrations?[b] (Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] PSEP control limit.

[PSDDA Tracking Number]

TABLE 13. CHECKLIST FOR AMPHIPOD MORTALITY BIOASSAY

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____ Batch ____ of ____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted[a]? (Y/N)_____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples_____

Interstitial salinities acceptable (i.e., ≥25 ‰)[c]? (Y/N)_____

If no, identify samples _____

FORMAT

Standard data report sheet

Number of survivors and percent mortality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N)_____

Water quality variables reported for each replicate? (Y/N)_____

QA/QC SAMPLES

Positive Control (not required by PSDDA)

Reference toxicant _____

Exposure concentrations _____

Organism response (LC50) _____

Dose response? (Y/N) _____

TABLE 13. (Continued)

QA/QC SAMPLES (continued)

Negative Control

Collection site _____

Total number _____

Mean mortality >10%?[c] _____

Analytical Replicates

Number per sample _____

Any <5 RPD?[c] _____

Mortality s.d. ≥15? [d] _____

Water Quality

Samples with temperature <14 or >16° C[c] _____

Samples with salinity <27 or >29 ‰[c] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤5 mg/L[e] _____

Reference

Collection site _____

Total number analyses _____

Mean mortality _____

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] PSEP control limit.

[d] PSEP guideline.

[e] General guideline.

TABLE 14. CHECKLIST FOR JUVENILE INFAUNA MORTALITY BIOASSAY

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____ Batch ____ of ____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Test species _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N)_____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples_____

FORMAT

Standard data report sheet

Number of survivors and percent mortality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N)_____

Water quality variables reported for each replicate? (Y/N)_____

QA/QC SAMPLES[c]

Positive Control (not required by PSDDA)

Reference toxicant _____

Exposure concentrations _____

Organism response (LC_{50}) _____

TABLE 14. (Continued)

QA/QC SAMPLES (continued)

Negative Control

Collection site _____

Total number _____

Mean mortality >10%?[d] _____

Analytical Replicates

Number per sample _____

Any <5 RPD?[d] _____

Reference

Collection site _____

Total number analyses _____

Mean mortality _____

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] Water quality conditions are not yet specified for this test.

[d] General guideline.

TABLE 15. CHECKLIST FOR SEDIMENT LARVAL BIOASSAY (SOLID PHASE)

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____ Batch ____ of ____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Test species _____ Exposure period _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N) _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples _____

FORMAT

Standard data report sheet

Number of larvae evaluated, percent mortality, and percent abnormality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N) _____

Water quality variables reported for each replicate? (Y/N) _____

QA/QC SAMPLES

Positive Control (not required by PSDDA)

Reference toxicant _____

Exposure concentrations _____

Organism response (EC_{50}) _____

TABLE 15. (Continued)

Negative Control (Seawater)[c]

Collection site _____

Total number _____

Mean abnormality >10%[d]? _____

Negative Control (Sediment)[c]

Collection site _____

Total number _____

Mean abnormality >10%[d]? _____

Analytical Replicates

Number per sample _____

Any <5 RPD?[d] _____

Water Quality

Crassostrea gigas

Samples with temperature <19 or >21° C[c] _____

Samples with salinity <27 or >29 ‰[c] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Mytilus edulis

Samples with temperature <12 or >14° C[e] _____

Samples with salinity <27 or >29 ‰[d] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Dendraster excentricus

Samples with temperature <11 or >13° C[e] _____

Samples with salinity <28 or >32‰[e] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

TABLE 15. (Continued)

Strongylocentrotus purpuratus	
Samples with temperature <11 or >13° C[e]	
Samples with salinity <28 or >32‰[e]	
Samples with pH <7 or >9[e]	
Samples with DO ≤4 mg/L[b]	
Reference	
Collection site	
Total number analyses	
Mean mortality	
Mean abnormality	

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] A control limit for mortality is presently not defined by PSDDA.

[d] PSEP control limit.

[e] General guideline.

TABLE 16. CHECKLIST FOR SEDIMENT LARVAL BIOASSAY (SUSPENDED PHASE)

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____ Batch ____ of ____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Test species _____ Exposure period _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N)_____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples_____

FORMAT

Standard data report sheet

Number of larvae evaluated, percent mortality, and percent abnormality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N)_____

Water quality variables reported for each replicate? (Y/N)_____

QA/QC SAMPLES

Positive Control (not required by PSDDA)

Reference toxicant _____

Exposure concentrations _____

Organism response _____

TABLE 16. (Continued)

Negative Control (seawater)

Collection site _____

Total number _____

Mean abnormality >10%[d]? _____

Negative Control (Sediment)[c]

Collection site _____

Total number _____

Mean abnormality >10%[d]? _____

Analytical Replicates

Number per sample _____

Any <5 RPD?[d] _____

Water Quality

Crassostrea gigas

Samples with temperature <19 or >21° C[c] _____

Samples with salinity <27 or >29 ‰[c] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Mytilus edulis

Samples with temperature <12 or >14° C[e] _____

Samples with salinity <27 or >29 ‰[d] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Dendraster excentricus

Samples with temperature <11 or >13° C[e] _____

Samples with salinity <28 or >32 ‰[e] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

TABLE 16. (Continued)

Strongylocentrotus purpuratus

Samples with temperature <11 or >13° C[e] _____

Samples with salinity <28 or >32 ‰[e] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Reference

Collection site _____

Total number analyses _____

Mean mortality _____

Mean abnormality _____

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] A control limit for mortality is presently not defined by PSDDA.

[d] PSEP control limit.

[e] General guideline.

3.10. CHECKLIST FOR MICROTOX™ BIOASSAY

The Microtox™ bioassay (saline extract) checklist is presented in Table 17. The Microtox™ organic extract bioassay is not used by PSDDA. QA1 measures for the Microtox™ bioassay include evaluation of holding conditions, holding times, positive and negative controls, and experimental conditions.

3.11. SUMMARY QA1 MATRIX

Summary matrices for chemical analyses and bioassays are presented in Tables 18 and 19, respectively. The QA measures listed in each table may not apply to all data categories listed. Qualitative and quantitative information from individual checklists is summarized in these checklists by PSDDA regulator during QA1 review. For example, "acceptable" could be used to represent conclusions regarding a QA1 characteristic that met all control limits. Similarly, "marginally acceptable, missing QA sample" would summarize a QA1 characteristic that the data reviewer found to be flawed but acceptable for dredging permit review. This information, along with the dredgers checklist, provides the basis for determining if the data are acceptable for permitting dredging operations.

[PSDDA Tracking Number]

TABLE 17. CHECKLIST FOR MICROTOX™ BIOASSAY

Project Name _____ Laboratory Report _____

Lab _____ Lab # _____ Batch ____ of ____

Responsible Technician _____

Reviewed by _____ Date checklist prepared _____

Date Sampled _____

Received by lab _____

Analysis began _____

Problems noted (e.g., deviations from prescribed methods, analytical problems)

_____

_____

All required documents submitted?[a] (Y/N) _____

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples _____

FORMAT

Standard data report sheet

Raw light emission data for each test series; 15-minute EC_{50}, 95-percent confidence limits, and evaluation of dose-responsiveness reported for each field sample, positive control, and negative control? (Y/N) _____

QA/QC SAMPLES

Positive Control

Reference toxicant _____

Exposure concentrations _____

Replicates/dilution _____

Organism response (EC_{50}) _____

TABLE 17. (Continued)

Reference

Blank ratio differences <0.02?[c] _____

Collection site for reference sediment _____

Total number _____

Dose-responsive? (Y/N) _____

Water Quality

Samples with temperature <14 or >16° C[d] _____

[a] Samples collected in field (laboratory QA samples excluded).

[b] PSDDA control limit.

[c] Beckman Instruments (1982).

[d] PSEP control limit.

TABLE 18. QA1 SUMMARY MATRIX - CHEMICAL VARIABLES

Matrix _____

QA1 Characteristic	Conventional Variables	Metals	Semivolatile Organic Chemicals	Volatile Organic Chemicals
% Complete Field				
Laboratory				
Units and Significant Figure				
Detection Limits				
Holding Conditions and Times				
Method Blank				
Standard Reference Material				
Replicates				
Matrix Spikes				

TABLE 19. QA1 SUMMARY MATRIX - BIOASSAYS

Matrix _____

QA1 Characteristic	Amphipod Mortality	Juvenile Infauna Mortality	Sediment Larval Test	Microtox
% Complete Field				
Laboratory				
Format				
Holding Conditions				
Positive Control				
Negative Control				
Replicates				
Experimental Conditions (Water Quality)				

4. GUIDANCE FOR QA1 DATA REVIEW AND INTERPRETATION

Guidance for QA1 data review and interpretation is presented schematically in Figure 4. The dredger's checklist and the chemistry and bioassay matrices summarize the information used to evaluate the acceptability of the data. The following matrix identifies eight criteria used for this evaluation and where they are applied:

	Dredgers Checklist	Chemistry Matrix	Bioassay Matrix
1. Compliance with sampling plan	X	X	X
2. Completeness of data set		X	X
3. Format		X	X
4. Precision		X	X
5. Chemical method blanks		X	
6 Accuracy of chemical analyses		X	
7. Bioassay controls			X
8. Bioassay testing conditions			X

Compliance with the sampling plan is determined from the dredger's checklist and the bioassay and chemistry matrix. Completeness, format, and precision apply to both chemical and bioassay data. Evaluation of blanks and accuracy is only conducted for chemical analyses. The evaluation of controls and experimental conditions only apply to bioassays. Guidance on the evaluation of these criteria is provided in the following sections. The conclusions of this evaluation are summarized in the PSDDA date review summary forms (Tables 20-22).

Rigid guidelines for passing or failing the data are not provided because QA1 is a simplified approach to data quality evaluation developed for a preliminary data review, not a comprehensive data evaluation. For data sets not meeting guidelines and control limits, the suitability assessments by PSDDA regulatory agencies will in most cases be based on best professional judgment and will include consultation with QA experts when appropriate.

Following criteria evaluation, a PSDDA regulator may reach one of three general conclusions: 1) the data set is within guidelines/control limits, 2) the data set is marginally outside guidelines/control limits for some variables, or 3) guidelines/control limits are violated severely by the data set. If the data set is within guidelines, the data set would pass PSDDA review. Other decisions made on the basis of data review are necessarily subjective. If the data set is marginally outside guideline/control limits, regulators may choose to accept the data for permitting purposes based on best professional judgment, or consult an expert to better evaluate why the data are outside guidelines/control limits and determine if their reasoning is sufficient to justify failing the data. This latter choice may require that a QA expert conduct a more thorough data review. If

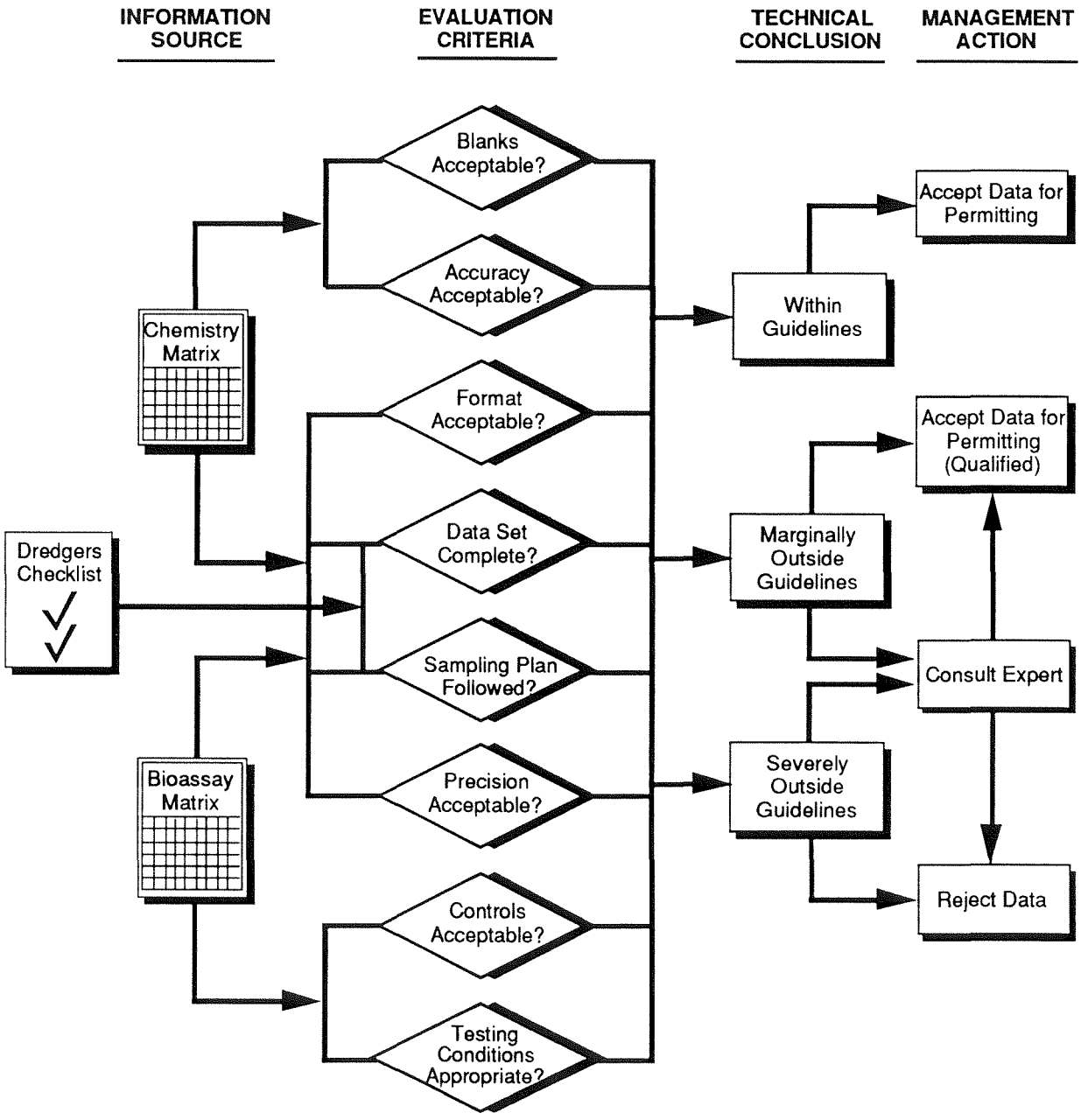

Figure 4. Guidance for QA 1 Data Review and Interpretation

TABLE 20. PSDDA DATA REVIEW SUMMARY: SEDIMENT CHEMISTRY

Data reviewer _____ Date reviewed _____

Sampling plan followed? _____

Data set complete? _____

Format acceptable? _____

Blanks acceptable? _____

Accuracy acceptable? _____

Precision acceptable? _____

Final Conclusions _____

TABLE 21. PSDDA DATA REVIEW SUMMARY: BIOASSAYS

Data reviewer _____ Date reviewed _____

Project name _____ PSDDA tracking # _____

Sampling plan followed? _____

Data set complete? _____

Format acceptable? _____

Precision acceptable? _____

Controls acceptable? _____

Testing conditions appropriate? _____

Final Conclusions _____

TABLE 22. PSDDA DATA REVIEW SUMMARY: BIOACCUMULATION

Data reviewer _____ Date reviewed _____

Project name _____ PSDDA tracking # _____

Sampling plan followed? _____

Data set complete? _____

Format acceptable? _____

Blanks acceptable? _____

Accuracy acceptable? _____

Precision acceptable? _____

Final Conclusions _____

the data set severely fails control limits/guidelines, PSDDA regulators may reject the data outright. The difference between marginal and severe exceedances of control limits is somewhat subjective, but can be illustrated by example. The control limit for PCB recovery in matrix spikes is 50 percent. If two matrix spikes were analyzed in a given data set and recoveries were 42 and 55 percent, this exceedance could be considered a marginal exceedance. If recoveries for both spikes were 5 percent, the exceedance should be considered severe and experts would probably recommend rejection of the data. Guidance for each evaluation criterion is provided in the following sections.

4.1. HAS THE SAMPLING PLAN BEEN FOLLOWED?

It is essential that the sampling plan be followed to ensure that the bioassay results accurately reflect environmental conditions and can be related directly to chemical measurements. Key considerations are that sediment samples are homogenized adequately, and that the bioassay subsample is taken from the same homogenate as the chemical subsample (called "synoptic collection"). If samples are not homogenized adequately, the bioassay results may reflect an unrepresentative portion of the field sample rather than the sample as a whole. If bioassay and chemical samples are not taken from the same homogenate, the relationship between observed sediment toxicity and measured chemical concentrations is less certain, and data may be unacceptable for incorporation in the sediment quality database. PSDDA does not require that chemical and bioassay samples always be collected synoptically. However, if this procedure is not followed, some elements of uncertainty exist as to how closely the chemical analyses and bioassays are related.

The use of appropriate sample handling and storage procedures is also essential to ensure that sediment toxicity is not altered between the times of field collection and laboratory analysis. For example, samples should not be frozen if storage at 4° C under nitrogen (strongly recommended for biological samples) is specified. In addition, sample holding time should not exceed the specified period. If handling and storage procedures are inappropriate, the measured sediment toxicity may not accurately reflect the true toxicity in the environment.

4.2. IS THE DATA SET COMPLETE?

One of the first steps in the QA process is to check whether the laboratory has provided results for all samples. If samples are missing, the laboratory's cover letter should provide an explanation for the absence. It is desirable that data sets contain all of the information necessary to conduct a QA/QC review and to use that information for permitting. Incomplete data sets are more likely to be a result of inadvertent omission from a data package than the irrevocable loss of a sample during analysis. Contact with the laboratory can typically resolve these problems.

The seriousness of missing information is variable, depending primarily upon the kind of information. For example, a missing replicate may not seriously affect the estimate of mean bioassay response if data from four other replicates are available. By contrast, the absence of control data (positive or negative) could cast doubt on the entire data set, because the responsiveness of the test organisms cannot be judged objectively. In general, the absence of QA/QC information is the most serious omission from the perspective of the entire data set. Other kinds of information generally relate to specific samples and usually have no influence on the remainder of the data set.

An important aspect of the assessment of completeness is whether or not all documentation needed for QA review is included in the laboratory's data package for chemical and biological analyses (see Appendix A). Completeness of QA documentation should be confirmed upon receipt of laboratory data even though some documentation may not be required immediately. Such checks will preclude inefficient requests for additional documentation long after the data have been generated.

4.3. IS THE FORMAT ACCEPTABLE?

4.3.1. Chemistry Data

Data reporting by the laboratory should conform to an appropriate format in terms of concentration units, number of significant figures, data qualifiers, and DL. For comparison to existing PSDDA screening levels and maximum levels, concentration units should be ppb dry-weight sediment for organic compounds (i.e., volatiles, A/B/N, PCB, and pesticides) and ppm dry-weight sediment for metals. Bioaccumulation results should be reported on an analogous wet-weight basis. Two significant figures are sufficient for comparison to sediment quality values. Although some laboratories report data to more than two significant figures, this practice is generally not warranted by the precision of routine environmental analysis (with the exception of certain metals analyses). Laboratory reporting of excessive significant figures is not a critical issue for QA1 analysis.

Data qualifiers should be clearly defined in the laboratory report sheets. For example, undetected compounds are indicated differently (e.g., < vs. U) by different laboratories. Undetected chemicals should be represented by DL in all samples.

During QA1 review, the review of DL focuses on regulatory concerns, that is, whether or not DL exceed PSDDA SL. Such exceedances of SL are problematic because they represent a "data gap" in available information on whether or not a sample requires biological testing. As a caveat, the acceptance of uniformly high DL by regulatory agencies may provide an incentive to perform insensitive chemical analyses that obscure moderate contamination. Although a comparison of sample DL to the technically based PSEP (1986) recommendations for DL is not a required element of QA1, it will yield useful information on data quality and is recommended.

DL are a critical aspect of data quality that is often overlooked. DL recommended by PSDDA/PSEP should be considered guidelines rather than firm criteria. In highly contaminated samples that require dilution or have high levels of interferences, DL for certain compounds may justifiably be higher than is desirable for cleaner samples. However, part of the justification for sample dilution and high DL is the presumption that high concentrations will be observed for at least a few compounds and will provide a basis for comparison to sediment quality values. For semivolatile organic compounds in particular, a range of DL can be expected, with the lowest DL for PAH with three or more rings (e.g., phenanthrene) and the highest DL for pentachlorophenol and other polar compounds, such as benzyl alcohol and benzoic acid.

Although validation of DL is beyond the range of QA1 data review, the reviewer should be wary of "optimistic" (extremely low) DL and DL that are very consistent among samples and chemicals. Consistent DL are themselves not a problem, but they sometimes indicate the assignment of default values that are not representative of the specific samples being analyzed.

4.3.2. Bioassay Data

Use of an appropriate format for data reporting facilitates both the review and use of a data set. Use of an inappropriate format generally does not influence the value of the data as long as the data set is complete. However, an inappropriate format can slow the review of the data set and, if sufficiently disruptive, may warrant reformatting prior to review and use.

4.4. ARE THE BLANKS ACCEPTABLE?

Blanks are used to assess laboratory contamination during sample preparation and analysis. Method blanks (preparation blanks for metals analyses) represent the net contamination of all stages of preparation and analysis. Laboratory contamination is of concern because it can result in "false positives" (i.e., erroneous reports of the compound as present in the sample) or overestimates of sample concentrations. QA control limits for blanks are typically based on the magnitude of blanks relative to DL and sample concentrations. Blanks are relevant to DL because they increase the level of background noise and interfere with the laboratory's ability to discern target chemicals in samples.

A complete evaluation of blanks (including blank correction, if appropriate) may not be within the scope of QA1 review because it would require a sample-by-sample evaluation of blank contributions relative to observed sample concentrations. Absolute control limits for blanks, which do not require a sample-by-sample analysis, are best suited for QA1 review. For organic compounds, the absolute PSEP control limits for blank contamination (expressed as total μg/blank sample) are most appropriate for QA1 review, whereas the relative control limits (expressed as a percent of sample concentrations) are appropriate for review when both the blank and sample data are computerized and can be easily manipulated. For metals, the absolute control limit under the EPA Contract Laboratory Program (CLP) and PSEP is that blank values must be less than DL. The relative control limit for metals is that detected results in samples can only be reported as detected when they are ≥ 5 times blank values.

The units in which laboratories report blanks are an important aspect of QA1 analysis of blanks. Blanks for organic compounds should be reported in total μg/blank sample, so they can be compared directly to absolute PSEP control limits. Blanks for metals in sediments are typically reported in units of mg/kg; these values can then be directly compared to DL. If blanks for a given chemical exceed an absolute control limit, the reviewer will have to decide whether to reject data for that chemical or to proceed with a more detailed analysis of blanks with relative (i.e., sample-by-sample) control limits. Among the PSDDA chemicals of concern, phthalates [especially bis(2-ethylhexyl)phthalate, di-n-octyl phthalate, and di-n-butyl phthalate] present the most frequent and severe contamination problems because of their presence in many plastic products.

4.5. IS THE ACCURACY ACCEPTABLE?

Accuracy is assessed by the analysis of matrix spikes and CRM (when available). Analytical recovery is an important aspect of data quality because poor recoveries will result in underestimates of chemical concentrations. High recoveries (e.g., >100 percent) may indicate problems with calibration or sample interferences. The matrix spike, a commonly analyzed indicator of accuracy, represents recovery of target compounds in a matrix that is similar to that of the samples being analyzed. Although QA1 review of accuracy only requires consideration of the percent recovery

of matrix spikes, it is useful to consider the concentration at which compounds were spiked. Spiking levels should be low enough to provide an estimate of recovery in the stated range of DL, but should be high enough to overwhelm the signal from compounds present in the unspiked sample. Preferably, spiking concentrations should be within an order of magnitude of DL. Laboratories sometimes spike at considerably higher levels, which can result in optimum recoveries that may not be representative of recovery at lower concentrations.

If problems with accuracy are encountered, decisions about data acceptability are best made after review of all available information for accuracy (including surrogate recoveries, which are sample-specific). If surrogate recovery data are computerized, they can be efficiently summarized during QA1 review and will provide valuable information about recovery for organic analyses. Data rejection is a severe action to be based on accuracy results but should be considered for compounds that are consistently not recovered (i.e., 0 percent recovery) or poorly recovered.

CRM for most target metals in sediments are available, but reference materials (certified or otherwise) for organic compounds in sediment and tissue are relatively rare. Although PSEP control limits do not strictly apply to performance on uncertified materials, these results can be useful and should be entered on checklists when available. They provide ancillary information to assess overall accuracy (i.e., in addition to matrix spikes and surrogate recovery).

4.6. IS THE PRECISION ACCEPTABLE?

4.6.1. Chemistry Data

Analytical replicates are used to evaluate the precision of the analytical method and instrumentation for the laboratory performing the analyses. Analytical precision is an expression of the variability of reported sample concentrations. Imprecision of reported laboratory results increases the uncertainty between comparisons of chemical data and sediment quality values. In routine CLP analyses for organic compounds, precision is assessed with matrix spike duplicates rather than with replicate analyses of unspiked samples. This approach ensures that target compounds will be present in the samples. Precision exceeding specified control limits indicates there is considerable uncertainty about the repeatability of the reported chemical results. However, rejection of data based upon precision exceeding control limits is a severe action.

If more than one set of replicates was performed, it is useful to examine the precision results for simple trends (e.g., was a certain compound or group of compounds consistently outside control limits, or did different compounds exceed control limits in different sets of replicates?). Consistent difficulties with certain compounds are generally of more concern than sporadic exceedances, which are more likely to indicate sample-specific difficulties (e.g., interferences, transcription errors) than consistent analytical difficulties.

4.6.2. Bioassay Data

For bioassays, replicate analyses are required for all samples. The Microtox™ test requires two replicate analyses per dilution, whereas the remaining tests require five replicate analyses per sample. At present, the only guideline available for bioassays is for the standard deviation of percent mortality in the amphipod mortality test (i.e., <15).

Unusually high variability among replicates acts to reduce the statistical power of comparisons with reference conditions. A reduction in power increases the chance that a "true" adverse effect will not be discriminated, and is therefore not environmentally protective. It is always advisable to review the variability (e.g., standard deviation of the mean) of the bioassay responses at all stations to check for outliers.

If a station is found with unusually high variability, the raw data for the individual replicate analyses should be inspected. If the high variability is the result of a single anomalous replicate, one might suspect that something unusual happened in the test chamber and that the replicate is not representative of the entire station. The replicate could be deleted from the station set and a new mean response could be calculated on the basis of the remaining replicates. If the high variability comes from variable responses in all replicates, one might suspect that test sediment was not homogenized sufficiently or that the bioassay was not run correctly.

It is helpful to consider the magnitude of the mean response when variability is high among all replicates. In general, high variability would not be expected when mean responses are either very high (i.e., because the sediment is very toxic) or very low (i.e., because the sediment is relatively uncontaminated). By contrast, high variability is sometimes found naturally when mean responses are moderate and may be the result of variable sensitivities among the individual test organisms.

4.7. ARE THE CONTROLS ACCEPTABLE?

Positive controls involve the exposure of representative test organisms to a reference toxicant, usually in a dilution series. These controls are used to ensure that the test organisms are suitably responsive to the effects of toxic chemicals. If the test organisms are not suitably responsive, the results of the bioassays may have little meaning. The results of positive control bioassays are usually reported as an LC_{50} or EC_{50} that can then be compared with the values found in other studies.

Negative controls involve the exposure of representative test organisms to clean seawater, sediment, or sediment extract. If possible, the negative control material should be collected from the native habitat of the test organisms. These controls are used primarily to ensure that the test organisms are suitably healthy for use in bioassays. In general, organisms are considered inappropriate for testing if the bioassay response in the negative controls is found in more than a small percentage (e.g., 10 percent) of the test population. Inadequate health could arise from natural stresses such as reproduction, low food supply, suboptimal water quality conditions, or from various experimental activities such as collection, holding, and acclimation. If organisms are not adequately healthy during testing, bioassay results may have little meaning.

4.8. ARE THE BIOASSAY TESTING CONDITIONS APPROPRIATE?

One of the main requirements of all bioassays is that they be conducted under controlled conditions so that the only variable that differs substantially among samples or studies is the one of interest (e.g., concentrations of a toxic chemical). In this manner, observed differences in bioassay responses among samples and studies can be attributed to the variable of interest with reasonable certainty. Therefore it is essential that the test conditions for each study (e.g.,

temperature, salinity, dissolved oxygen, pH) be maintained within the limits specified in the bioassay protocol. Failure to maintain test conditions within these limits can jeopardize comparisons among samples and studies, because any observed differences in bioassay responses may be partly related (to an unknown degree) to the variable test conditions.

4.9. ARE THE BIOASSAY SAMPLE SIZES ADEQUATE?

The sediment larval tests involving bivalves and echinoderms are different from most of the other bioassays because the sample size is not controlled at the beginning of testing. Instead, sample sizes for abnormality determinations depend on how many organisms survive the test. In some cases, mortality can be very high, leaving few larvae for abnormality determinations. If mortality is 100 percent, the sample size of survivors drops to zero, and the abnormality endpoint cannot be assessed.

One method of addressing this problem is to specify that a minimum number of larvae (e.g., 40) be evaluated for each replicate test. Due to uncertainties in the number of larvae surviving each test, multiple samples from each test chamber should be archived so they can be analyzed if the initial sample does not contain a sufficient number of larvae for abnormality determinations. In cases where mortality is high, additional laboratory time may have to be spent reading slides until the minimum sample size is achieved. If mortality is so high that the minimum number of larvae cannot be achieved with reasonable effort, the value determined for abnormality could be qualified as being based on a suboptimal number of replicates. An alternative approach would be to combine the mortality and abnormality endpoints under the assumption that all organisms that died must have exhibited abnormal development prior to death.

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GLOSSARY

Accuracy—The closeness of a measured or computed value to its true or expected value. Accuracy is used to denote only systematic error or bias.

Amphipods—Small shrimp-like crustaceans (e.g., sand fleas). Many live on the bottom, feed on algae and detritus, and serve as food for marine species. Amphipods are commonly used in laboratory bioassays to test the toxicity of sediments because they are relatively sensitive to chemical toxicity.

Analyte—The specific component measured in a chemical analysis.

Apparent Effects Threshold (AET)—The sediment concentration of a contaminant above which statistically significant biological effects would always be expected.

Area Ranking—The designation of a dredging area relative to its potential for having sediment chemicals of concern. Rankings range from "low" potential to "high" potential, and are used to determine the intensity of dredged material evaluation and testing that might be required.

Batch—Usually refers to the number of samples that can be prepared or analyzed at one time. A typical commercial batch size is 20 samples for extraction of organic compounds.

Bioaccumulation—The accumulation of chemicals in the tissues of an organism (e.g., certain chemicals in food eaten by a fish tend to accumulate in its liver or other tissues).

Bioassay—A laboratory test used to evaluate the toxicity of a material (commonly sediments or wastewater) by exposing organisms to the material under controlled conditions and measuring their behavioral, physiological, or lethal responses.

Biota—The animals and plants that live in a particular area.

Blank-Corrected—The concentration of a chemical in a sample adjusted for the concentration of that chemical in the method blank carried through the procedure concurrently with the sample.

Bottomfish—Fish (e.g., English sole) that live on or near the bottom of a body of water in close contact with the sediment.

Bulk Chemical Analyses—Chemical analyses performed on an entire sediment sample, without separating water from the solid material in a sample.

Calibration—The systematic standardization of either the response of instruments used for measurements or the chemical separation achieved by a laboratory cleanup procedure.

Certified Reference Material (CRM)—A reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body. A standard reference material (SRM) is a certified reference material issued by the National Bureau of Standards.

Coefficient of Variation—The standard deviation expressed as a percentage of the mean.

Confined Disposal—A disposal method that isolates the dredged material from the environment. Confined disposal may be in aquatic, nearshore, or upland environments.

Contaminant—A chemical or biological substance in a form or in a quantity that can harm aquatic organisms, consumers of aquatic organisms, or users of the aquatic environment.

Contaminated Sediment

Technical Definition: A sediment that contains measurable levels of contaminants.

Management or Common Definition: A sediment that contains sufficient concentration(s) of chemicals to produce unacceptable adverse environmental effects and thus require restriction(s) for dredging and/or disposal of dredged material (e.g., is unacceptable for unconfined, open-water disposal or conventional land/shore disposal, requiring confinement).

Control Limit—Defines the minimum quality of data as measured by some indicator (e.g., recovery) required to assume that the system or method is performing as expected. Exceedance of a control limit triggers action by the laboratory to correct the problem before data are reported. Control limits can be required (mandatory) or recommended.

Conventional Variables—Sediment parameters and characteristics other than chemical contaminants that have been routinely measured in assessing sediment quality. These include sulfides, organic carbon, etc.

Corrective Action—Measures taken to remove, adjust, remedy, or counteract a malfunction or error so that a standard or required condition is met.

Detection Limit—The smallest concentration or amount of some component of interest that can be measured by a single measurement with a stated level of confidence. In practice, detection limits can be determined by different methods in different laboratories and are not always assigned a statistical level of confidence.

Disposal Site—The bottom area that receives discharged dredged material, encompassing and larger than the target area and the disposal area.

Dredged Material—Sediments excavated from the bottom of a waterway or water body.

Dredged Material Management Unit—The maximum volume of dredged material for which a decision on suitability for unconfined, open-water disposal can be made. Management units are typically represented by a single set of chemical and biological test information obtained from a composite sample. Management units are smaller in areas of higher chemical contamination concern (see **Area Ranking**).

Dredger—Private developer or public entity (e.g., federal or state agency, port, or local government) responsible for funding and undertaking dredging projects and not necessarily the dredging contractor who physically removes and disposes of dredged material (see below).

Dredging—Any physical digging into the bottom of a water body. Dredging can be done with mechanical or hydraulic machines and is performed in many parts of Puget Sound for the maintenance of navigation channels that would otherwise fill with sediment and block ship passage.

Dredging Contractor—Private or public (e.g., U.S. Army Corps of Engineers) contractor or operator who physically removes and disposes of dredged material for the dredger (see above).

Dry Weight—The weight of a sample based on percent solids. The weight after drying in an oven.

Duplicate Analysis—A second analysis made on the same (or identical) sample of material to assist in the evaluation of measurement variance.

Evaluation Procedures Work Group (EPWG)—The PSDDA work group that is developing chemical and biological testing and test evaluation procedures for dredged material assessment.

Field Sample—The term field sample includes all samples collected from a discrete coordinate in a dredging volume. A composite may be prepared from two or more field samples.

Gas Chromatography (GC)—An instrumental technique used to separate a complex mixture into its component compounds by partitioning the compounds between a mobile gaseous phase (under pressure) and a stationary solid or liquid phase.

Gas Chromatography/Electron Capture Detection (GC/ECD)—An instrumental technique useful for the determination of organic compounds containing halogens (e.g., chlorine).

Gas Chromatography/Flame Ionization Detection (GC/FID)—An instrumental technique useful for the detection of organic compounds that can be converted to ions during exposure to a flame.

Gas Chromatography/Mass Spectroscopy (GC/MS)—An instrumental technique useful for breaking organic compounds into characteristic fragments that can be used to determine the original structure of the compound.

Gel Permeation Chromatography (GPC)—A cleanup procedure used to remove interfering biological macromolecules from sample extracts.

Gravid—Having eggs, such as female crabs carrying eggs.

Habitat—The specific area or environment in which a particular type of plant or animal lives. An organism's habitat provides all of the basic requirements for life. Typical Puget Sound habitats include beaches, marshes, rocky shores, bottom sediments, mudflats, and the water itself.

High Pressure (or High Performance) Liquid Chromatography (HPLC)—An instrumental technique used to separate a complex mixture into its component compounds by partitioning the compounds between a mobile liquid phase (under high pressure) and a stationary solid phase.

Hydrocarbon—An organic compound composed of carbon and hydrogen. Petroleum and its derived compounds are primarily hydrocarbons.

Injection Internal Standards—A standard added to a sample extract just prior to instrumental analysis. This standard is used to determine the actual percent recovery of the surrogate spike compounds. When the isotope dilution technique is not used, the injection internal standard is also used to quantify compounds of interest in the sample relative to standards.

Isotope Dilution Technique—A technique for quantification of organic compounds that uses a large number of stable isotopically labeled compounds (i.e., compounds for which some hydrogen atoms have been replaced with deuterium, or some carbon-12 atoms have been replaced with carbon-13) spiked in the sample before sample extraction to correct for compound losses during sample workup. The labeled compounds are analogs of the compounds of interest and behave similarly.

Matrix—The sample material in which the chemicals of interest are found (e.g., water, sediment, tissue).

Matrix Spike—An analysis conducted by adding a known amount of chemicals of interest to an actual sample (i.e., matrix), usually prior to extraction or digestion, and then carrying the spiked sample through the analytical procedure. The final matrix spike results are reduced by the amount of each chemical found in a replicate analysis of the sample conducted without spikes. A comparison of these results with the known concentration of spike added to the sample enables an evaluation of the effect of the particular sample matrix on the recovery of compounds of interest.

Metals—Metals are 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 unnatural amounts by human activities.

Method Blank—A measure of the contribution of analytes from all laboratory sources external to the sample. The method blank value is determined by proceeding through all phases of extraction and analysis with no addition of sample.

Method Spike—A method blank to which a known amount of surrogate standards and analytes (compounds of interest) has been added.

Microtox™—A laboratory bioassay using luminescent bacteria and measuring reductions in light production as the test endpoint, often used to assess toxicity of saline or organic sediment extracts.

Noise—The electronic signal intensity attributed to instrument "background" or electronic current from chemical interferents (i.e., any part of an electrical signal that cannot be related in a known way to the electronic current from a target compound).

Overdepth Material—Dredged material removed from below the dredging depth needed for safe navigation. Though overdepth is incidentally removed due to dredging equipment precision, its excavation is usually planned as part of the dredging project to ensure proper final water depths. Common overdepth is 2 feet below the necessary dredging line.

Oxygen Demanding Materials—Materials such as food waste and dead plant or animal tissue that use up dissolved oxygen in the water when they are degraded through chemical or biological processes. Chemical and biological oxygen demand (COD and BOD) are different measures of how much oxygen a particular substance demands.

Parameter—A quantifiable or measurable characteristic of something (e.g., height, weight, sex, and hair color are all parameters that can be determined for humans). Water quality parameters include temperature, pH, salinity, dissolved oxygen concentration, and many others.

Permit—A written warrant or license, granted by an authority, allowing a particular activity to take place. Permits required for dredging and disposal of dredged material include the U.S. Army Corps of Engineers Section 404 permit, the Washington State Department of Fisheries Hydraulics Permit, the city or county Shoreline Development Permit, and the Washington Department of Natural Resources Site Use Disposal Permit.

Pesticide—A general term used to describe any substance, usually chemical, used to destroy or control organisms (pests). Pesticides include herbicides, insecticides, algicides, and fungicides. Many of these substances are manufactured and are not naturally found in the environment. Others are natural toxins that are extracted from plants and animals.

pH—The degree of acidity or basicity of a solution, which is a function of hydronium ion concentration. A pH of less than 7.0 indicates an acidic solution, and a pH greater than 7.0 indicates a basic solution. The pH of water influences many of the types of chemical reactions that occur in it.

Phase I—The PSDDA study is divided into two 3-year overlapping phases. Phase I covers the central area of Puget Sound including Seattle, Everett, and Tacoma. Phase I began in April 1985.

Phase II—The PSDDA study is divided into two 3-year overlapping phases. Phase II covers north and south Puget Sound (including Olympia, Bellingham, and Port Angeles)--the areas not covered by Phase I. Hood Canal is not being considered for location of a disposal site. Phase II began in April 1986.

Polychlorinated Biphenyls (PCB)—A group of manufactured organic chemicals, including 209 different but closely related compounds (congeners) made up of carbon, hydrogen, and chlorine. If released to the environment, they persist for long periods of time and can concentrate in food chains. The manufacture and use of PCB are regulated by EPA under the Toxic Substances Control Act.

Polycyclic (Polynuclear) Aromatic Hydrocarbon (PAH)—A class of organic compounds, some of which are persistent and carcinogenic. These compounds are formed from the combustion of organic material and are ubiquitous in the environment. PAH are commonly formed by forest fires and by the combustion of fossil fuels. PAH often reach the environment through atmospheric fall-out, highway runoff, and oil discharge.

Polytetrafluoroethylene (PTFE)—The generic chemical name for materials such as Teflon, a registered trademark of the duPont Corporation.

Precision—The degree of mutual agreement characteristic of independent measurements as the result of repeated application of a method under specified conditions. It is concerned with the closeness of results.

Preparation Blank (reagent blank, method blank)—An analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

Priority Pollutant—Toxic pollutants defined by EPA in 1976 that are the primary subject of regulation of the Clean Water Act. A list of these substances can be found in the Code of Federal Regulations Volume 40, Section 401.15.

Protocol—A compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

PSDDA Tracking Number—An 11 digit number assigned by the Corps to identify individual dredging surveys. The code includes information that represents project/survey, testing phase (i.e., PC or FC), calendar year, month, and agency from which the data originated.

Puget Sound Water Quality Authority (PSWQA)—An agency created by the Washington state legislature in 1985 and tasked with developing a comprehensive plan to protect and enhance the water quality of Puget Sound. The PSWQA adopted its first plan in January 1987.

Quality Assurance (QA)—The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Control (QC)—The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

Quantification—The determination or expression of the number or amount of a variable.

Reconstructed Ion Chromatogram—A graphical display of the total ionization current resulting from all mass fragments detected over time during a mass spectral analysis. The chromatogram can be used to indicate the relative composition of components in the sample mixture analyzed by GC/MS.

Recovery—The amount of a chemical detected in a sample extract at the end of a procedure relative to the total amount present in a sample before the procedure was begun. Also, the amount of a chemical detected in a sample relative to the amount added (i.e., spike) or known to be present (i.e., in a naturally derived certified reference material). Recovery is usually expressed as a percentage.

Relative Percent Difference (RPD)—Difference of two measurements x_1 and x_2, divided by the mean of the measurements, multiplied by 100.

Replicate—One of several identical experiments, procedures, or samples. Duplicate is a special case of replicates consisting of two samples or measurements.

Reproducibility—The ability to produce the same results for a measurement. Often measured by calculation of relative percent difference or coefficient of variation.

Resection—The surgical removal of tissue from an organism during sampling (dissection is the sectioning of tissues within the organism, but does not entail removal of the tissues).

Response Factor—Generally, the ratio of the amount (mass) of a substance to a measurement of its response over time measured by the detector of an analytical instrument. The ratio of response factors for a chemical and a surrogate spike in a sample, or a chemical in a sample and a standard calibration is used to quantify the concentration of chemicals in a sample.

Sediment—Mineral and organic material suspended in or settling to the bottom of a liquid, such as the sand and mud that make up much of the shorelines and bottom of Puget Sound. Sediment input to Puget Sound comes from natural sources, such as erosion of soils and weathering of rock, or anthropogenic sources, such as forest or agricultural practices or construction activities. Certain contaminants tend to collect on and adhere to sediment particles. The sediments of some areas around Puget Sound contain elevated levels of contaminants.

Semivolatile Organic Compounds—Organic compounds with moderate vapor pressures that can be extracted from samples using organic solvents and analyzed by gas chromatography. In this document, semivolatile organic compounds include the EPA acid/base/neutral compounds, pesticides and PCB, as well as numerous other neutral and organic acid compounds of regional interest (e.g., carbazole, retene, coprostanol, 4-methylphenol).

Sensitivity—Capability of a method or instrument to discriminate between samples having differing concentrations of a chemical. The degree to which an instrument responds to low concentrations of a chemical.

Significant Difference—A quantitative determination of the probability that two measurements of the same parameter are different, given the variability of the measurements.

Significant Figure—A figure that remains to a number or decimal after the zeros to the right or left are cancelled.

Spike—The addition of a known amount of a substance to a sample.

Standard—A substance or material, the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property of a sample. In chemical measurements, a standard often describes a solution of chemicals, commonly prepared by the analyst, to establish a calibration curve or the analytical response function of an instrument.

Standard Reference Material (SRM)—See Certified Reference Material.

Strong Acid Digestion (SAD)—SAD refers to a digestion technique that employs nitric acid, hydrogen peroxide, and hydrochloric acid to extract metals from sediments. Because it is a milder digestion than TAD, SAD may not extract all metals from sediments.

Study Sample—The term study sample applies to all samples remaining after compositing has been conducted. Study samples include composite samples prepared from two or more field samples and field samples that were not composited.

Surrogate Spike Compound—A known amount of a compound that has characteristics similar to that of a compound of interest, added to a sample prior to extraction. The surrogate compound can be used to estimate the recovery of chemicals in the sample. These compounds are also called "recovery internal standards".

Target Compounds—The chemicals of interest in a sample that can be quantified relative to response factors of reliable standards (in contrast to tentatively identified compounds).

Tentatively Identified Compounds—Chemicals identified in a sample on the basis of mass spectral characteristics held in common with a reference mass spectra of a known chemical. These compounds cannot be more confidently identified unless a reliable standard of the compound is obtained and is confirmed to co-elute with the tentatively identified compound and generate similar mass spectra using the same GC/MS.

Total Acid Digestion (TAD)—TAD refers to a digestion technique that employs hydrofluoric acid and aqua regia to extract metals from sediments. The TAD digestion technique is recommended by PSDDA.

Unconfined, Open-Water Disposal—Discharge of dredged material into an aquatic environment, usually by discharge at the surface, without restrictions or confinement of the material once it is released.

Volatile Organic Compounds—Organic compounds with high vapor pressures that tend to evaporate readily from a sample. In this document, volatile organic compounds are the 29 EPA priority pollutants considered as volatiles (e.g., benzene).

Volatile Solids—The material in a sediment sample that evaporates at a given high temperature.

Warning Limit—In Puget Sound programs, a value either above or below which data returned by a laboratory are subjected to qualification before inclusion in a regional database. The principle is identical to that of a control limit, but is less stringent and serves as a warning that the system or method may become out of control.

Water Quality Certification—Approval given by Washington Department of Ecology acknowledging the compliance of a discharge with Section 401 of the Clean Water Act.

Wet Weight—The weight of a sample aliquot including moisture (undried).

APPENDIX A

Required Laboratory Documentation

INTRODUCTION

Considerable documentation must be obtained from laboratories for a complete data review. Although some of this documentation is not necessary for QA1 review, it is needed for QA2 review. The following lists, excerpted from PSEP protocols, specify documentation that must be included in laboratory data packages for chemical and bioassay analyses. For the sake of efficiency, complete documentation must be confirmed upon receipt of the laboratory package, so additional requests will not be required during a subsequent QA2 review.

CHEMICAL VARIABLES

ORGANIC COMPOUNDS

The following documentation is needed for organic compounds:

- A cover letter referencing or describing the procedure used and discussing any analytical problems
- Reconstructed ion chromatograms for GC/MS analyses for each sample
- Mass spectra of detected target compounds (GC/MS) for each sample and associated library spectra
- GC/ECD and/or GC/flame ionization detection chromatograms for each sample
- Raw data quantification reports for each sample
- A calibration data summary reporting calibration range used [and decafluorotriphenylphosphine (DFTPP) and bromofluorobenzene (BFB) spectra and quantification report for GC/MS analyses]
- Final dilution volumes, sample size, wet-to-dry ratios, and instrument detection limit
- Analyte concentrations with reporting units identified (to two significant figures unless otherwise justified)
- Quantification of all analytes in method blanks (ng/sample)
- Method blanks associated with each sample
- Recovery assessments and a replicate sample summary (laboratories should report all surrogate spike recovery data for each sample; a statement of the range of recoveries should be included in reports using these data)
- Data qualification codes and their definitions.

METALS

For metals, the data report package for analyses of each sample should include the following:

- Tabulated results in units as specified for each matrix in the analytical protocols, validated and signed in original by the laboratory manager
- Any data qualifications and explanation for any variance from the analytical protocols
- Results for all of the QA/QC checks initiated by the laboratory
- Tabulation of instrument and method detection limits.

All contract laboratories are required to submit metals results that are supported by sufficient backup data and quality assurance results to enable independent QA reviewers to conclusively determine the quality of the data. The laboratories should be able to supply legible photocopies of original data sheets with sufficient information to unequivocally identify:

- Calibration results
- Calibration and preparation blanks
- Samples and dilutions
- Duplicates and spikes
- Any anomalies in instrument performance or unusual instrumental adjustments.

BIOASSAYS

Amphipod Mortality Test

The following data should be reported by all laboratories performing this bioassay:

- Water quality measurements during testing (e.g., dissolved oxygen, temperature, salinity, pH)
- Daily emergence for each beaker and the 10-day mean and standard deviation for each treatment
- 10-day survival in each beaker and the mean and standard deviation for each treatment
- Interstitial salinity values of test sediments
- 96-hour LC_{50} values with reference toxicants
- Any problems that may have influenced data quality.

Juvenile Infauna Mortality Test

The following data should be reported by all laboratories performing this bioassay:

- Water quality measurements during testing (e.g., dissolved oxygen, temperature, salinity, pH)
- 10-day survival in each beaker and the mean and standard deviation for each treatment
- 96-hour LC_{50} values with reference toxicants
- Any problems that may have influenced data quality.

Bivalve Larval Test (Solid Phase)

The following data should be reported by all laboratories performing this bioassay:

- Water quality measurements at the beginning and end of testing (e.g., dissolved oxygen, temperature, salinity, pH)
- Individual replicate and mean and standard deviation data for larval survival after 48 hours
- Individual replicate and mean and standard deviation data for larval abnormalities after 48 hours
- 48-hour LC_{50} and EC_{50} values with reference toxicants
- Any problems that may have influenced data quality.

Bivalve Larval Test (Suspended Phase)

The following data should be reported by all laboratories performing this bioassay:

- Water quality measurements at the beginning and end of testing (e.g., dissolved oxygen, temperature, salinity, pH)
- Individual replicate and mean and standard deviation data for larval survival after 48 hours
- Individual replicate and mean and standard deviation data for larval abnormalities after 48 hours
- 48-hour LC_{50} and EC_{50} values with reference toxicants
- Any problems that may have influenced data quality.

Microtox Test (Saline Extract)

The following data should be reported by all laboratories performing this bioassay:

- Percent decrease in luminescence for each concentration of supernatant (e.g., saline sediment extract) tested, including blanks
- Determination of a significant dose-response relationship by least-squares regression of percent decrease in luminescence on the logarithm of sample dilution
- Determination of EC_{50} values and 95-percent confidence limits for the reference toxicant
- Any problems that may have influenced data quality.

APPENDIX B

Example Checklists

INTRODUCTION

The example checklists in this appendix are slightly modified versions of checklists submitted to PSDDA; however, the dredger's checklist and chemistry checklists were based on different projects than the bioassay checklists. These example checklists are intended to clarify the information requested in the checklists but are not intended to be "ideal" examples; in fact, not all aspects of these checklists are in strict compliance with PSDDA requirements. The example summary matrices (one each for chemistry and bioassays) are based on information in the relevant example checklists. The format for filling out summary matrices is somewhat less rigid than that for the checklists. The matrices should include notes that the reviewer considers pertinent to the data quality with respect to PSDDA requirements.

[PSDDA Tracking Number]

TABLE 5. DREDGER'S CHECKLIST

Project Name Waffle Marina Applicant M. Jones

Location S. Sound, near Shelton Date February 14, 1989

Area Rank(s) Moderate

Partial characterization (PC) or full characterization (FC)? FC

Total volume of sediment to be dredged (attach diagram) 100,000 cu yd

SAMPLES COLLECTED

	Chemistry	Bioassays	Bioaccumulation
# Stations	25	25	
# Field samples[a]	25	25	
# Study samples[b]	7	7	
Shipboard storage conditions			

ANALYTICAL STRATEGY

Chemistry only ____ Synoptic chemistry and biology ✓

Biology only ____ Chemistry 1st, biology 2nd ____

TARGET VARIABLES

	Chemistry	Bioaccumulation
All PSSDA chemicals of concern	Yes	
Additional chemicals	Butyltins	
Deleted chemicals	None	

FIELD COLLECTION

What positioning method was used? (describe) Miniranger 3

Station positioning information attached? Yes

Sampling strategy (attach diagram) Yes

Sampling device Vibra corer

Were chemistry/bioassay samples homogenized prior to subsampling? Yes

Were corresponding chemistry and bioassay subsamples taken from the same composite? Yes

[a] Field samples includes all samples collected from discrete coordinates in a dredging volume.
[b] Study samples includes all samples remaining after compositing has been conducted.

[PSDDA Tracking Number]

TABLE 6. CHECKLIST FOR CONVENTIONAL VARIABLES IN SEDIMENT

Project Name Olympic Harbor 11/88 Laboratory Report Technical Memorandum 270

Lab ABC, Inc. Lab # C13512

Responsible Technician Chemist A

Reviewed by Reviewer A Date checklist prepared 1/10/89

Date: Sampled November 7-9, 1988

Received by lab Same

Analysis began November 11, 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)
None

COMPLETENESS AND HOLDING CONDITIONS

	TOC	TVS	Total Sulfides	Ammonia	Total Solids	Grain Size Distribution
Method (identify)	(Combustion) PSEP	PSEP	PSEP	Plumb (1981)	PSEP	PSEP Sieve & pipet
# Samples submitted	25	25	25	25	25	25
# Samples analyzed	25	25	25	25	25	25

Holding conditions acceptable? (Y/N) Yes

(see final page of checklist for guidelines)

If no, identify samples

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures Yes

Sample detection limits provided, when applicable (total sulfides, ammonia) Yes

Qualifiers defined (e.g., U = undetected)
Footnoted in tables

TABLE 6. (Continued)

QA/QC SAMPLES

Method Blank

TOC Total # 2

Frequency 8%
(minimum 1 per 20 samples)[a]

Amount detected in blank <1 µg/g dry weight
(no PSEP control limit)

Certified Reference Materials

TOC Total # Not analyzed

Frequency _____
(minimum 1 per survey)[a]

CRM used _____

Within 95% confidence interval? _____
(not a PSEP control limit)

Analytical Replicates

	TOC	TVS	Total Sulfides	Ammonia	Total Solids	Grain Size Distribution
Total #	25	25	25	25	25	25
Frequency (minimum 1 triplicate per 20 samples)[a]	2 tripl.	1 tripl.	0	2 tripl.	1 tripl.	1 tripl.

[a] Recommended by PSEP (1986).

TABLE 6. (Continued)

RECOMMENDED SAMPLE SIZES, CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR SEDIMENT CONVENTIONAL VARIABLES (PSEP 1986)

Variable	Minimum Sample Size (grams)[a]	Container[b]	Preservation	Maximum Holding Time
Particle size	100-150[c]	P,G	Cool, 4° C	6 months[d]
Total solids	50	P,G	Freeze	6 months[d]
Total volatile solids	50	P,G	Freeze	6 months[d]
Total organic carbon	25	P,G	Freeze	6 months[d]
Total sulfides	50	P,G	Cool, 4° C, 1N zinc acetate	7 days[d]
Ammonia	20	P,G	Cool, 4° C (minimize air contact, keep field moist)	7 days[e]

[a] Recommended field sample sizes for one laboratory analysis. If additional laboratory analyses are required (e.g., replicates), the field sample size should be adjusted accordingly.

[b] P = polyethylene; G = glass.

[c] Sandy sediments require larger sample sizes than do muddy sediments.

[d] This is a suggested holding time. No EPA criteria exist for the preservation of this variable.

[e] This holding time is recommended by Plumb (1981).

TABLE 7. CHECKLIST FOR METALS IN SEDIMENT

Project Name Olympia Harbor Laboratory Report Technical Memorandum 270

Lab ABC, Inc. Lab # M7357

Responsible Technician Chemist C

Reviewed by Reviewer B Date checklist prepared 2/15/89

Date Sampled November 7-9, 1988

Received by lab Same

Analysis began November 28, 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)
None

All required documents submitted[a]? (Y/N) Yes

Digestion procedure [Total Acid Digest (TAD) or Strong Acid Digest (SAD)] TAD

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted 25 # Samples analyzed 25

Holding conditions acceptable? (Y/N) [6 months frozen for metals except mercury; 28 days frozen (in glass) for mercury] Yes

If no, identify samples

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures Yes

Qualifiers defined (e.g., U = undetected)
See footnotes in tables

Sample detection limits (DL) provided for each analyte? (Y/N) Yes

TABLE 7. (Continued)

QA/QC SAMPLES

Preparation Blank

Total # 7

Frequency[b] ≥ 1/batch of 10
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals observed above detection limits in one or more blanks[c]

None

Certified Reference Materials

Total # 12

Frequency[b] 2/batch of 10
(minimum 5% or 1 per batch, whichever is more frequent)[c]

CRM used NBS 1646, 1645, and NRCC-MESS-1, BCSS-1, PACS-1

Chemicals outside 80-120% recovery[c] (outside 95% C.I.)

Zn in NBS 1645 Zn, As, Hg in PACS-1

Hg in NBS 1646

(for chemicals without certified values, use matrix spike results)

Analytical Replicates

Total # 6 sets of triplicates

Frequency[b] 1/batch of 10
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Samples/chemicals with >20% relative percent difference (RPD) or coefficient of variation (CV)[c]

None

Matrix Spikes

Total # Not analyzed

Frequency[b]
(minimum 5% or 1 per batch, whichever is more frequent)[c]

Chemicals with recovery outside 75-125%[c]

TABLE 7. (Continued)

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

Antimony _____ Arsenic _____ Cadmium _____

Copper _____ Lead _____ Mercury _____

Nickel _____ Silver _____ Zinc _____

(see Table 2)

Preparation Blanks (Relative blank contamination)

Are sample results <5 times blank values in any samples? (Y/N) _____

If yes, identify elements and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤5 samples should be as follows: CRM > analytical replicate > matrix spikes.

[c] PSEP control limit.

[PSDDA Tracking Number]

TABLE 8. CHECKLIST FOR SEMIVOLATILE ORGANIC COMPOUNDS IN SEDIMENT

Project Name Olympia Harbor Laboratory Report Technical Memorandum 270

Lab ABC, Inc. Lab # SV1258

Responsible Technician Chemist D (extraction/cleanup); Chemist E (GC)

Reviewed by Reviewer D Date checklist prepared 1/10/89

Date Sampled November 7-9, 1988

Received by lab Same

Analysis began December 19, 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)

None

All required documents submitted[a]? (Y/N) Yes

Analytical method MacLeod et al. (1985)

COMPLETENESS AND HOLDING CONDITIONS

	# Samples Submitted	# Samples Analyzed
A/B/N	25	25
Pesticides/PCB	25	25

Holding conditions acceptable? (Y/N) (1 year for frozen sediment)

Yes

If no, identify samples

Extract holding times acceptable? (Y/N) Yes (< 40 days)

If no, identify samples

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures Yes

Qualifiers defined (e.g., U = undetected)

See footnotes in table

TABLE 8. (Continued)

Sample detection limits (DL) provided for each analyte? (Y/N) Yes

QA/QC SAMPLES

Method Blank

Total # 3

Frequency 1/extraction batch of 10 samples
(minimum 1/per extraction batch)[b]

Chemicals detected above 5 μg total (for phthalates) and 2.5 μg total[b] (for other organic compounds; lower levels may be appropriate for pesticides and PCBs)

None

Certified Reference Materials

Total # 1

Frequency 1/25
(<50 samples - 1 per set of samples submitted to lab; >50 samples - 1 per 50 samples analyzed)[b]

CRM used Duw III (not certified)

Chemicals outside 95% confidence interval (for certified values)[b]

None outside

Analytical Replicates

Total # 1 triplicate

Frequency 2/25
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicate for minimum of 5% total replication)[b]

Samples/chemicals with >100% RPD or CV[b]

None

Matrix Spikes (not required for A/B/N if isotope dilution used)

Total # 2 (for analytes not in reference material)

Frequency[c] 2/25
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 5% of total samples)[b]

Chemicals with <50% recovery[b]

None for phthalates spiked

TABLE 8. **(Continued)**

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

A/B/N (PAH) _____ A/B/N (phenols, benzoic acid, benzyl alcohol) _____ A/B/N (other) _____

PCB _____ Pesticides _____

(see Table 2)

Surrogate Recovery (A/B/N)

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10 percent recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50 percent recovery[b] (also identify samples)

_____ _____

_____ _____

Surrogate Recovery (Pesticides/PCBs)

Were surrogates added to all samples?[b] (Y/N) _____

Identify samples with <50 percent surrogate recovery[b]

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

For target compounds other than phthalates, was blank contamination >5 percent of any sample concentrations?[b] (Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

TABLE 8. (Continued)

For phthalates, was blank contamination >50 percent of any sample concentration?[b]

(Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] For batches of 5 samples or less, the minimum QA checks should be a blank and the analysis of a CRM (and matrix spikes for any analytes not certified in the CRM). In general, the priority of QA checks for batches of ≤ 5 samples should be as follows: CRM > analytical replicate > matrix spikes.

[c] PSEP control limit.

TABLE 9. CHECKLIST FOR VOLATILE ORGANIC COMPOUNDS IN SEDIMENT

Project Name Olympia Harbor Laboratory Report Technical Memorandum 270

Lab ABC, Inc. Lab # V1103

Responsible Technician Chemist F

Reviewed by Reviewer G Date checklist prepared 1/10/89

Date Sampled November 7-9, 1988

Received by lab Same

Analysis began November 14, 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)
Hexachloroethane not quantified

All required documents submitted?[a] (Y/N) No data for hexachloroethane

Analytical method Purge-and-trap GC/FID

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted 25 # Samples analyzed 25

Holding conditions acceptable? (Y/N) (14 days at 4° C) Yes

If no, identify samples

FORMAT

Standard data report sheet

Concentrations in proper units and significant figures Yes

Qualifiers defined (e.g., U = undetected)
See footnotes in tables

Sample detection limits (DL) provided for each analyte? (Y/N) Yes

TABLE 9. (Continued)

QA/QC SAMPLES

Method Blank

Total # 4

Frequency ≥ 1 per batch of 10 samples
(minimum 1 per batch or 1 per 12-hour shift, whichever is more frequent)[b]

Chemicals detected above 2.5 μg total in one or more blanks[b]

None

Analytical Replicates

Total # 1 triplicate

Frequency 2/25
(<20 samples - 1 per set of samples submitted to lab; ≥20 samples - 1 triplicate and additional duplicates for 5% replication overall)[b]

Samples/chemicals with >100% RPD or CV[b]

None

Matrix Spikes (not required if isotope dilution used)

Total # 2

Frequency 2/25
(<20 samples - 1 per set of samples submitted; ≥20 samples - 5% overall)[b]

Chemicals with <50% recovery[b]

None

NOTE: The following information will be filled out by PSDDA personnel rather than the laboratory.

Detection Limits

Did any DL exceed SL? (Y/N) _____

If yes, detection limits exceeding SL (identify samples)

Volatiles _____

(see Table 2)

TABLE 9. (Continued)

Surrogate Recovery

Were surrogates added to all samples?[b] (Y/N) _____

Was the isotope dilution technique used? (Y/N) _____

If yes, identify compounds with <10 percent recovery (also identify samples)

_____ _____

_____ _____

If no, identify compounds with <50 percent recovery[b]

_____ _____

_____ _____

Method Blanks (Relative blank contamination)

Was blank contamination >5 percent of any sample concentrations?[b] (Y/N) _____

If yes, identify compounds and samples

_____ _____

_____ _____

[a] See Appendix A for list.

[b] PSEP control limit.

[PSDDA Tracking Number]

TABLE 13. CHECKLIST FOR AMPHIPOD MORTALITY BIOASSAY

Project Name Mutiny Bay Laboratory Report Tech Memo 971

Lab Clamshell, Inc. Lab # 3718-02 Batch 1 of 1

Responsible Technician G.O. Duck

Reviewed by Bob Barker Date checklist prepared 25 December 1988

Date Sampled 7 November 1988

Received by lab 30 November 1988

Analysis began 6 December 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)

None

All required documents submitted[a]? (Y/N) Yes

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted 12 + 1 ref + 1 control/# Samples analyzed 12 + 1 ref + 1 control/

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] Yes

If no, identify samples —

Interstitial salinities acceptable (i.e., ≥25 ‰)[c]? (Y/N) Yes

If no, identify samples —

FORMAT

Standard data report sheet

Number of survivors and percent mortality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N) Yes

Water quality variables reported for each replicate? (Y/N) Yes

QA/QC SAMPLES

Positive Control (not required by PSDDA)

Reference toxicant Na PCP

Exposure concentrations 0.21, 0.30, 0.64, 0.93, 0.145, 0.442 mg/L

Organism response (LC50) 0.40 mg/L

Dose response? (Y/N) Yes

TABLE 13. (Continued)

QA/QC SAMPLES (continued)

Negative Control

Collection site: West Beach, Whidbey Is.

Total number: 1

Mean mortality >10%?[c]: No (8%)

Analytical Replicates

Number per sample: 5

Any <5 RPD?[c]: No

Mortality s.d. ≥15?[d]: No

Water Quality

Samples with temperature <14 or >16° C[c]: Yes, two (16.5, 16.8)

Samples with salinity <27 or >29 ‰[c]: Yes, one (30)

Samples with pH <7 or >9[e]: None

Samples with DO ≤5 mg/L[e]: None

Reference

Collection site: Carr Inlet

Total number analyses: 1

Mean mortality: 10%

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] PSEP control limit.

[d] PSEP guideline.

[e] General guideline.

TABLE 14. CHECKLIST FOR JUVENILE INFAUNA MORTALITY BIOASSAY

Project Name Muting Bay Laboratory Report Tech Memo 971

Lab Clamshell, Inc. Lab # 3718-02 Batch 1 of 1

Responsible Technician G.O. Duck

Reviewed by Bob Barker Date checklist prepared 25 December 1988

Date Sampled 7 November 1988

Received by lab 30 November 1988

Analysis began 6 December 1988

Test species Neanthes arenaceodentata

Problems noted (e.g., deviations from prescribed methods, analytical problems)

None

All required documents submitted?[a] (Y/N) Yes

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted 12 + 1 ref + 1 control # Samples analyzed 12 + 1 ref + 1 control

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] Yes

If no, identify samples —

FORMAT

Standard data report sheet

Number of survivors and percent mortality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N) Yes

Water quality variables reported for each replicate? (Y/N) Yes

QA/QC SAMPLES[c]

Positive Control (not required by PSDDA)

Reference toxicant None

Exposure concentrations NA

Organism response (LC_{50}) NA

TABLE 14. (Continued)

QA/QC SAMPLES (continued)

Negative Control

Collection site: West Beach, Whidbey Is.

Total number: 1

Mean mortality >10%?[d]: Yes (15%)

Analytical Replicates

Number per sample: 5 samples/treatment, 10 organisms/jar

Any <5 RPD?[d]: No

Reference

Collection site: _____

Total number analyses: _____

Mean mortality: _____

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] Water quality conditions are not yet specified for this test.

[d] General guideline.

[PSDDA Tracking Number]

TABLE 15. CHECKLIST FOR SEDIMENT LARVAL BIOASSAY (SOLID PHASE)

Project Name Mutiny Bay Laboratory Report Tech Memo 971

Lab Clamshell, Inc. Lab # 3718-02 Batch 1 of 1

Responsible Technician G.O. Duck

Reviewed by Bob Barker Date checklist prepared 25 December 1988

Date Sampled 7 November 1988

Received by lab 30 November 1988

Analysis began 6 December 1988

Test species Crassostrea gigas Exposure period 48 hours

Problems noted (e.g., deviations from prescribed methods, analytical problems)

None

All required documents submitted?[a] (Y/N) Yes

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted 12 + 1ref + 1 control + 1 seawater # Samples analyzed Same

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] Yes

If no, identify samples —

FORMAT

Standard data report sheet

Number of larvae evaluated, percent mortality, and percent abnormality reported for each replicate (including field samples, positive controls, and negative controls)? (Y/N) Yes

Water quality variables reported for each replicate? (Y/N) Yes

QA/QC SAMPLES

Positive Control (not required by PSDDA)

Reference toxicant Na PCP

Exposure concentrations 1,000, 560, 320, 180, 100 (ppb)

Organism response (EC_{50}) 382 ppb (95% Ch: 180-560)

TABLE 15. (Continued)

Negative Control (Seawater)[c]

Collection site: Clean Bay

Total number: 1

Mean abnormality >10%[d]? No (8%)

Negative Control (Sediment)[c]

Collection site: West Beach, Whidbey Is.

Total number: 1

Mean abnormality >10%[d]? No (7%)

Analytical Replicates

Number per sample: 5

Any <5 RPD?[d] No

Water Quality

Crassostrea gigas

Samples with temperature <19 or >21° C[c]: Two (22°, 23°)

Samples with salinity <27 or >29 ‰[c]: None

Samples with pH <7 or >9[e]: One (6.5)

Samples with DO ≤4 mg/L[b]: Two (3.8, 3.7)

Mytilus edulis

Samples with temperature <12 or >14° C[e]:

Samples with salinity <27 or >29 ‰[d]:

Samples with pH <7 or >9[e]:

Samples with DO ≤4 mg/L[b]:

Dendraster excentricus

Samples with temperature <11 or >13° C[e]:

Samples with salinity <28 or >32‰[e]:

Samples with pH <7 or >9[e]:

Samples with DO ≤4 mg/L[b]:

TABLE 15. (Continued)

Strongylocentrotus purpuratus

Samples with temperature <11 or >13° C[e] _____

Samples with salinity <28 or >32‰[e] _____

Samples with pH <7 or >9[e] _____

Samples with DO ≤4 mg/L[b] _____

Reference

Collection site _____

Total number analyses _____

Mean mortality _____

Mean abnormality _____

[a] See Appendix A for list.

[b] PSDDA control limit.

[c] A control limit for mortality is presently not defined by PSDDA.

[d] PSEP control limit.

[e] General guideline.

[PSDDA Tracking Number]

TABLE 17. CHECKLIST FOR MICROTOX™ BIOASSAY

Project Name Mutiny Bay Laboratory Report Tech Memo 971

Lab Clamshell, Inc. Lab # 3718-02 Batch 1 of 1

Responsible Technician G.O. Duck

Reviewed by Bob Barker Date checklist prepared 25 December 1988

Date Sampled 7 November 1988

Received by lab 30 November 1988

Analysis began 6 December 1988

Problems noted (e.g., deviations from prescribed methods, analytical problems)

None

All required documents submitted?[a] (Y/N) Yes

COMPLETENESS AND HOLDING CONDITIONS

Samples submitted _____ # Samples analyzed _____

Holding conditions acceptable? (Y/N) (4° C in nitrogen atmosphere ≤6 weeks)[b] _____

If no, identify samples _____

FORMAT

Standard data report sheet

Raw light emission data for each test series; 15-minute EC_{50}, 95-percent confidence limits, and evaluation of dose-responsiveness reported for each field sample, positive control, and negative control? (Y/N) Yes

QA/QC SAMPLES

Positive Control

Reference toxicant Phenol

Exposure concentrations 0, 7.3, 14.5, 29.1, 58.1 mg/L

Replicates/dilution 2

Organism response (EC_{50}) 28 (25-33)

TABLE 17. (Continued)

Reference

Blank ratio differences <0.02?[c] Yes

Collection site for reference sediment Sequim Bay

Total number 8

Dose-responsive? (Y/N) No

Water Quality

Samples with temperature <14 or >16° C[d] None

[a] Samples collected in field (laboratory QA samples excluded).

[b] PSDDA control limit.

[c] Beckman Instruments (1982).

[d] PSEP control limit.

TABLE 18. QA1 SUMMARY MATRIX - CHEMICAL VARIABLES

Matrix Sediment

QA1 Characteristic	Conventional Variables	Metals	Semivolatile Organic Chemicals	Volatile Organic Chemicals
% Complete Field	100%	100%	100%	100%
Laboratory	100%	100%	100%	100% (No data for hexachloroethane)
Units and Significant Figure	OK	OK	OK	OK
Detection Limits	OK	OK	OK (Generally low)	OK
Holding Conditions and Times	OK	OK	OK	OK
Method Blank	OK (TOC)	OK	OK	OK
Standard Reference Material	Not analyzed (TOC)	Some chemicals outside 95% C.I. (esp. Zn, Hg); check if 80-120% recovery	OK (Duwam. Riv. III)	(Not applicable)
Replicates	Total sulfides not replicated	OK	OK	OK
Matrix Spikes	—	Not analyzed, but high frequency of SRM and other QA samples	OK (only spiked phthalates)	OK

TABLE 19. QA1 SUMMARY MATRIX - BIOASSAYS

Matrix Sediment Bioassays

QA1 Characteristic	Amphipod Mortality	Juvenile Infauna Mortality	Sediment Larval Test	Microtox
% Complete Field				
Laboratory	100%	100%	100%	100%
Format	OK	OK	OK	OK
Holding Conditions	OK	OK	OK	OK
Positive Control	OK	None	OK	OK
Negative Control	OK (8%)	Failed (15%)	OK Mortality = 20% Abnormality = 7%	NA
Replicates	OK (5)	OK (5)	OK (5)	OK (2 per dilution)
Experimental Conditions (Water Quality)	2 samples exceed temperature; 1 sample exceeds salinity	NA	2 samples exceed temperature; 1 sample exceeds pH; 2 samples exceed DO	OK