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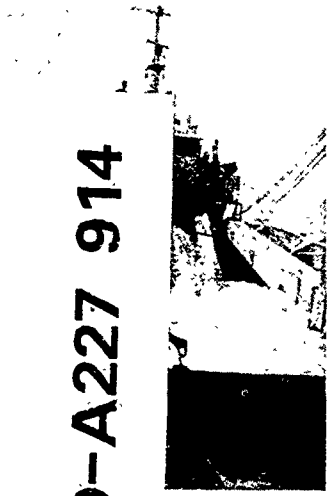

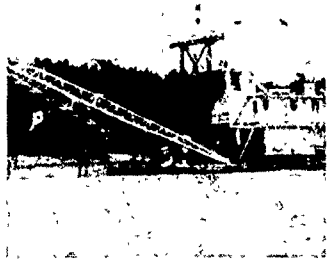

ENVIRONMENTAL LABORATORY

DREDGING OPERATIONS TECHNICAL SUPPORT PROGRAM

TECHNICAL REPORT D-90-10

GUIDANCE FOR CONTRACTING BIOLOGICAL AND CHEMICAL EVALUATIONS OF DREDGED MATERIAL

by

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Environmental Laboratory

DEPARTMENT OF THE ARMY
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Contracting is a cost-effective method for increasing the capability of the Corps of Engineers to conduct dredged material evaluations. Test data generated by a contractor are used as an integral part of the decision-making process by the Corps and the US Environmental Protection Agency regarding the suitability of dredged material for disposal. Therefore, conscientious contract management is vital to ensure that the generated data are of the highest quality, are obtained in a competent fashion, and can be defended against legal-scientific challenge.

This report provides a general outline of the various types of contractors, guidance on a variety of activities required for management of chemical and biological contracts, and quality assurance/quality control techniques specifically for dredged and fill material. This report will provide project managers with procedures that will produce data that are reliable and can be used as the basis for important regulatory decisions.

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PREFACE

This work was conducted by the Environmental Laboratory (EL), US Army Engineer Waterways Experiment Station (WES), as part of the Dredging Operations Technical Support (DOTS) Program. The DOTS Program is sponsored by the Dredging Division of the Headquarters, US Army Corps of Engineers (HQUSACE), and is managed by the EL through the Environmental Effects of Dredging Programs (EEDP). Dr. Robert M. Engler was Program Manager for the EEDP; Mr. Thomas R. Patin was the DOTS Program Manager. Mr. Joseph Wilson, HQUSACE, was the Technical Monitor.

This document was coordinated by Mr. Paul Lang in 1981 and was revised/updated by Mr. Thomas C. Sturgis of the Aquatic Processes and Effects Group (APEG), Ecosystem Research and Simulation Division (ERSD), EL. Ms. Ann B. Strong, Chief of the Analytical Laboratory Group, Environmental Engineering Division (EED), EL, and Ms. Alfreda B. Gibson of the Contaminant Mobility and Regulatory Criteria Group, ERSD, EL, assisted in the revision.

The report was prepared under the general supervision of Dr. Thomas L. Hart, Chief, APEG; Mr. Donald L. Robey, Chief, ERSD; Dr. Raymond L. Montgomery, Chief, EED; and Dr. John Harrison, Chief, EL. The report was edited by Ms. Jessica S. Ruff of the WES Information Technology Laboratory.

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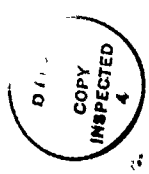

CONTENTS

CONVERSION FACTORS, NON-SI TO SI (METRIC) UNITS OF MEASUREMENT

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
gallons (US liquid)	3.78541	cubic decimeters
inches	25.4	millimeters

GUIDANCE FOR CONTRACTING BIOLOGICAL AND CHEMICAL EVALUATIONS OF DREDGED MATERIAL

PART I: INTRODUCTION

Background

1. A major function of the US Army Corps of Engineers is to maintain navigable waterways, which may require the removal of 290 million cubic meters of material annually from the Nation's navigation system (lakes, harbors, etc.). However, the potential presence of contaminants in some sediment has created concern that disposal of dredged material may have an adverse ecological effect on aquatic and terrestrial biota (Francingues et al. 1985). This concern has led to the regulation of dredged material for environmental protection under Section 404 of the Clean Water Act (CWA) (Public Law (PL) 92-500, as amended) and Section 103 of the Marine Protection, Research, and Sanctuaries Act (PL 92-532, as amended). Section 404 authorizes the Secretary of the Army, acting through the Corps, to issue permits for the discharge of dredged or fill material into waters of the United States in accordance with the Section 404(b)(1) Guidelines (subsequently referred to as the Guidelines). These Guidelines are intended to restore and maintain the chemical, physical, and biological integrity of waters of the United States through the control of discharges of dredged or fill material.

2. Section 404(b)(2) allows the Corps to issue permits otherwise prohibited by the Guidelines, based on an overriding consideration of the economics of anchorage and navigation.

3. The Guidelines require compliance with several conditions prior to allowing disposal of dredged material in waters of the United States. Compliance requires the avoidance of "unacceptable adverse effects" to the aquatic environment. The Guidelines specify four conditions of compliance ("restrictions on discharge" per 40 CFR 230.10) (Engler et al. 1986), as stated below.

a. There is no other practicable alternative that would have less adverse impact on the aquatic environment.

b. The disposal will not result in violations of applicable water quality standards after consideration of dispersion and dilution (40 CFR 230.10(b)(1)), toxic effluent standards, or marine

sanctuary requirements, nor will it jeopardize the continued existence of threatened or endangered species.

c. The disposal will not cause or contribute to significant degradation of the waters of the United States.

d. All appropriate and practicable steps have been taken to minimize potential adverse impacts of the discharge on the aquatic environment.

4. Findings for compliance with condition b are based in large part on Section 401 of the CWA, which allows the individual states to establish State water quality standards. The findings of compliance with condition c are based, in part, on "evaluation and testing" of the proposed dredged material (Subpart G of the Guidelines). The assessment provided by Subpart G is used to determine the potential for substantive adverse effects of dredged material disposal on the aquatic environment (factual determinations required by Part 230.11). According to the Guidelines (40 CFR 230.61), specific evaluation procedures, including chemical and biological tests to determine compliance with the Guidelines and State water quality standards, are furnished by the Corps as the permitting authority (US Environmental Protection Agency/ US Army Corps of Engineers 1977). The Implementation Manual (USEPA/USACE 1977) describes acceptable procedures for conducting tests required by PL 92-532, and similar procedures are being used in fresh water in relation to the CWA. However, the implementation manual does not provide guidance for the selection and management of contractors to conduct these tests.

5. With increasing workload and hiring restrictions imposed on the Corps of Engineers (CE), contracting has become a cost-effective method for increasing the capability of the CE to conduct dredged material evaluations. Although contractors can be used to increase the CE capabilities, care must be taken to ensure that the appropriate contracting mechanism is used, high-quality assurance and quality control standards are met, and the data generated by different contractors are comparable. Unfortunately, contract guidance for quality assurance (QA) and quality control (QC) specific for dredged material has not been available. This report sets forth guidelines specifically for chemical and biological evaluation of dredged or fill material. The report was developed based on elements of existing QA and QC plans and past CE experience in contract management. It is our hope that through the use of the information contained in this report, CE personnel will be able to avoid many of the pitfalls experienced by others in the past.

6. Several contracting methods discussed in this document are applicable to CE needs or preferences. The contracting process consists of a series of sequential actions, which begins with the determination that the CE need can best be met by use of a contract and culminates with the satisfactory performance of a contract to meet that need. However, the most important aspect of the contracting process (which includes contract development, contractor selection, and contract management) is contract management. Poor management can negate or invalidate an entire study. It is imperative that the manager selected for the specific contract(s) be involved in the development of the scope of work and throughout the contracting procedures. Contract management must be proactive rather than reactive.

7. A major concern of both the CE and US Environmental Protection Agency is the reliability of the test data upon which proposed project evaluations are based. The CE should require all laboratories providing analytical services to maintain a quality control program that is realistic and experimentally achievable. A sound quality control program is most effective when it represents the objectives of all individuals involved. The user of the data must appreciate the quality and have confidence in the data. Laboratory management should be dedicated to achieving high-quality goals and committed to providing the resources to obtain them.

Objectives

8. The objectives of this report are to provide quality assurance guidelines that will increase the confidence level in biological and chemical results and to establish guidelines that the CE can use in selecting contractors to perform chemical and biological evaluations of dredged or fill material.

Organization of Report

9. This guidance has been prepared in five parts: Introduction, Contracting, Quality Assurance Considerations Applicable to Both Biological and Chemical Procedures, Laboratory Selection and Contract Management for Dredged Material Bioassay and Bioaccumulation Studies, and Management of Chemical Analysis Contracts. Part II discusses the different types of contractors,

contracting methods, and contract management. Parts III, IV, and V discuss quality control and quality assurance in selecting contractors and managing biological and chemical analysis contracts. While chemical and biological concerns are addressed separately, it is perfectly acceptable to award one contract that includes both types of analyses to a qualified contractor.

10. For convenience, bibliographic citations are listed at the conclusion of each part of the report.

References

Engler, R. M., et al. 1986. "Corps of Engineers' Procedures and Policies on Dredging and Dredged Material Disposal (The Federal Standard)," Environmental Effects of Dredging Technical Note EEDP-04-8, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

Francingues, N. R., Jr., Palermo, M. R., Lee, C. R., and Peddicord, R. K. 1985. "Management Strategy for Disposal of Dredged Material: Contaminant Testing and Controls," Miscellaneous Paper D-85-1, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

US Environmental Protection Agency/US Army Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. 1977. "Ecological Evaluation of Proposed Discharge of Dredged Material Into Ocean Waters; Implementation Manual for Section 103 of Public Law 92-532 (Marine Protection, Research, and Sanctuaries Act of 1972)" (2d printing, April 1978), US Army Engineer Waterways Experiment Station, Vicksburg, MS.

PART II: CONTRACTING

Types of Contractors

General

11. The various types of contractors are described in general terms in this part with the thought of providing at least an outline to allow the CE to choose the type of contractor who offers the greatest probability of success for the particular need at hand. Note that individual evaluation of each potential contractor is necessary. None of the following characteristics attributed generally to types of contractors can automatically be assumed to apply to any particular contractor of that type.

Profit and nonprofit enterprises

12. Since the academic institutions are generally nonprofit entities, they can sometimes offer lower costs. This may result in increases in cost-effectiveness, although this varies greatly and cannot be assumed. The private firm, which operates at a profit, generally is highly competitive financially, which sometimes results in a tendency to maximize gains on a particular project.

13. Academic institutions tend to have an orientation toward basic research, which may influence the areas into which they direct their resources. For example, if a university consistently engages in activities considered to have little educational or research value, such as routine surveys, they may encounter difficulties with their governing bodies. An exception to this would be institutions that recognize the benefits students can obtain from hands-on experience gained through the opportunity to participate in surveying studies. Firms operating for profit are rarely concerned with education. They will, within their resources, do anything, ranging from basic research to routine surveying and testing.

14. Academic institutions and their personnel are accustomed to utilizing grant procedures. Under these procedures there is often, but not always, broad leeway in meeting deadlines, schedules, and producing results in a timely fashion. Private firms generally do not have this outlook. They are accustomed to contracts whereby a particular end product is clearly specified with the results being delivered within a specific time frame. In fact,

private firms may insist that a "loose" contract be tightened so as to limit their overall responsibility and liability.

15. Professors at academic institutions are sometimes constrained by academic schedules. However, during the nonacademic portion of the year, faculty are often highly mobile to go anywhere they are needed to carry out any number of tasks that could not be done during the academic year. Private firms often are the epitome of flexibility. The project or program manager, as the case may be, is often able to draw upon a common staffing pool on a short-term or sometimes long-term basis to meet his need. This flexibility will at times be a distinct advantage. At other times the common staffing pool may be preempted by a project manager other than the one being dealt with.

16. Responsibility in academic institutions may be very diffuse. Responsibility is often legally vested in a president or research office. However, the work is usually carried out by a Principal Investigator who is at the same time a faculty member. The contract is usually not signed with the faculty member, who is under no obligation, other than that imposed by the institution, to deliver results. If the Principal Investigator performs poorly for the Corps but is a faculty member with tenure, little recourse may be possible to correct the situation. This is often not the case with private firms, where one deals with a project or program manager who has direct project responsibility. Should this program or project manager not perform satisfactorily, it is possible that the individual may be warned of being relieved of project responsibility or even of being fired. Such a warning may result in improved work performance by the project manager.

17. Recognition of the general characteristics of profit and nonprofit enterprises and their implications for satisfactory project completion by both technical and procurement personnel is extremely helpful in selecting a contractor, and in interacting with that contractor throughout the contract period.

Government agencies

18. Interagency agreements are a recognized means of getting Government requirements satisfied. These agreements are not to be used for the purpose of avoiding competitive procurement procedures. They are permitted and encouraged by the Economy Act as a means of getting the Government's requirements satisfied in the most economic, timely, and efficient way available.

19. Heads of Staff Offices or their designees are generally responsible for managing interagency agreements along with planning, drafting, and submitting proposed agreements for review and approval authority. Designated project managers are usually responsible for seeing that the terms and conditions of the agreement are adhered to and for periodically reporting the status of the agreement to proper authority.

20. While interagency agreements have not been used extensively in the past to evaluate dredged material, their use is not precluded. However, time and manpower constraints on other agency programs will often result in this option being infeasible. In addition, an agency's programs can be reprioritized after entering into an interagency agreement, thus affecting the effort allocated to the agreement work. However, interagency agreements to evaluate dredged material might be extremely beneficial to the Corps in that hands-on familiarity by other agencies would likely improve interagency communication and understanding.

Contracting Methods

Sealed bidding

21. The Federal Government's preferred method of contracting is through sealed bidding. This is accomplished through Solicitation for Bids from all qualified sources of services deemed necessary by the Contracting Officer to ensure full and open competition. Contracts resulting from sealed bidding are normally from-fixed-price.

22. While sealed bidding has been used by the CE for biological and chemical evaluations of dredged material, its use is not without potential technical problems. It is virtually impossible to specify every analytical detail required or quantify the expertise required, etc. Thus, it is difficult to determine if all bidders are equally technically qualified. Even so, there is an administrative tendency within the CE to use the sealed bid process to obtain environmental data, rather than the Request for Proposals (RFP) process.

Negotiation

23. When sealed bidding is not considered appropriate because of a need to consider factors other than low price in selection of a contractor, solicitation is made by RFP to those organizations considered capable of

performing the required work. It is required by statute that procurements be competitive to the maximum practical extent, whether the procurement is being obtained by sealed bidding or negotiating procedures.

24. Evaluation of dredged material by negotiated contract is considered acceptable and has certain advantages. It allows prospective contractors to propose use of acceptable techniques that are not familiar to the Corps. This can result in technical improvements and cost reductions in analysis methods. Reviewing contractors' proposals also allows an objective evaluation of their expertise by weighing all technical and nontechnical considerations to select the most qualified contractor.

Small and Disadvantaged Business competition considerations

25. Small Business. The Small Business Act grants the Small Business Administration powers to assist and endorse contracting with small businesses. Thus, small businesses are assisted in obtaining a fair share of the procurement dollars spent by the Federal Government.

26. Corps personnel should consult with District procurement and Small Business specialists to ascertain what Small Business considerations might be feasible for the dredged material evaluation program.

27. Minority and Small Disadvantaged Business. Within the Small Business Program is the Small Disadvantaged Business Program, commonly known as the 8(a) program. Basically, this program encourages minority-owned businesses to participate in contracting with the Federal Government for its supplies and service needs by forming a partnership with the Small Business Administration. This contract is awarded via 8(a) authority.

Small purchases

28. Small purchases are identified as purchases of supplies and services, the aggregate amount of which does not exceed $25,000. These purchases are normally procured by means of a simplified set of procedures which reduce the administrative costs for these smaller value procurements.

29. Requirements totaling more than $25,000 will not be broken down into several purchases merely for the purpose of permitting negotiation under the small purchases procedures. Competition is required in obtaining small purchases over $2,500, and soliciting three to five sources is generally considered acceptable.

30. Dredged material evaluation by small purchases is obviously limited by the cost constraint.

Federal supply schedules

31. To promote greater efficiency and economy in Government procurement programs, the General Services Administration establishes contracts for common use classes of services. These contracts are summarized in Federal Supply Schedules (FSS) that contractors and the supplies and services that are available from them. To promote maximum economy in Government procurement, purchasing officers should use these contracts at all times when the delivery requirements can be met. Even though the stated FSS delivery date exceeds that required, the Contracting Officer should check with the contractor since the stated delivery dates are conservative, and frequently the contractor can deliver faster if required.

32. The FSS should be periodically reviewed to determine if biological or chemical evaluations of dredged material can be conducted by this method.

Labor surplus and disaster area firms

33. It is also appropriate to point out that firms located in labor surplus or disaster areas, or firms which, under a Government contract, furnish articles that are substantially manufactured in such areas, may receive preferential treatment in Government procurement. In certain instances, this preference overrides Small Business preferences. In general, the Labor Surplus Area Program of a Government agency operates in much the same way as the Small Business Program. Procurement regulations require that firms located in such areas be placed on an appropriate bidders list and be given an opportunity to submit quotations on procurements for which they are qualified. Even when bidders lists are rotated, and only a portion of a list is solicited for each procurement, all labor surplus area concerns on the overall list published by the Department of Labor must be solicited for procurements of more than $25,000.

34. Corps personnel should contact District procurement experts to decide what labor surplus and disaster area considerations might be feasible to the dredged material evaluation program.

Broad Agency Announcement

35. The Provisions of the Competition in Contracting Act of 1984 (PL 98-369) as implemented in the Federal Acquisition Regulation provide for the issuance of a Broad Agency Announcement (BAA) as a means of announcing

potential interest for basic research. This announcement must be general in nature, identify the areas of research interest, include criteria for selecting proposals, and solicit the participation of all offerors capable of satisfying the Government needs. The proposals submitted under the BAA will be subject to peer or scientific review. Proposals that are selected for award are considered to be the results of full and open competition and in full compliance with the provisions of PL 98-369.

36. The BAA is published annually by the US Army Engineer Waterways Experiment Station (WES) and remains in effect until superseded by the latest edition. The WES encourages Historically Black Colleges and Universities (HBCUs), Minority Institutions (MIs), Small Business concerns, and Small Disadvantaged Business concerns to submit research proposals for consideration. Some research will be set aside for participation only by HBCUs and MIs, to facilitate achievement of the goals established by Section 1207 of PL 99-661 and Section 806 of PL 100-180. Proposals from US Government facilities (Districts, Divisions, etc.) will not be accepted.

Task Orders

37. Task Order (TO) contracts are also called indefinite delivery and/or requirements contracts. These contracts allow flexibility in permitting tasks to be accomplished when needed, and reduce procurement lead-time by allowing project managers use of contractors more than once without initiating the contracting process, provided the task was included in the original scope of work. Task orders are awarded through RFP from qualified sources of services deemed necessary by the contracting officer to ensure full and open competition. The RFP includes the estimated cost of equipment and materials, manpower, and travel. Manpower cost is computed by multiplying the total number of employees times the hours required to complete a task: this is then multiplied by the salary rate of each employee. Educational background and experience dictate employee(s) salary rate. Material and equipment that are deemed necessary to complete a task are calculated. Travel cost associated with the anticipated task is also calculated, and may include distances to and from test sites and project-related meetings between the contractor and project manager. The costs for completion of all tasks are added; profit and overhead, if appropriate, are then calculated.

38. The selection of a TO contractor is based primarily on factors other than cost. Generally, the factors that are used to select a contractor

are experience and qualification. However, if funds for procurement are limited, the cost proposed may be quite important in source selection. Points are assigned as weights to the various factors, which may then be subdivided, providing partial weight to each subfactor. It is not essential that the total number of points equal 100; however, this is commonly the case. An example (purely for illustration) is provided below:

Factors		Points
1. Qualifications		35
a. Specific experience	10	
b. Technical organization	9	
c. Quality of performance (past history)	6	
d. Educational level (personnel)	10	
2. Cost		5
a. Salaries (scientific versus administrative)	5	
3. Equipment		35
a. Special facilities/equipment	20	
b. Analytical capability	10	
c. Simplicity of project design	5	
4. Time of completion		25

39. The points are added, and the contractor is given a composite rating. The contractor with the highest composite rating is usually awarded the contract with an estimated amount shown, but with no obligation of money from the procurement office. At this point a specific scope of work must be developed. (Appendix A shows an example scope of work.) The scope of work must be neither so narrow as to unduly restrict the contractor effort nor so broad as to permit the contractor to explore areas having only peripheral interest and relationship to the required work. It must clearly define the contractor's obligation and should therefore be sufficiently definitive to protect the Government's interest.

40. Descriptive ratings may be used in the evaluation of proposals ("unsatisfactory," "poor," "average," "good," "excellent," or perhaps "no information provided"). Descriptive ratings can be combined with numerical ratings. The purpose of establishing numerical factors is to provide a working tool. The quantitative expression of evaluation criteria helps

analysis and reveals deficiencies. However, the numerical ratings are only as good as the underlying analysis that is made when they are established and the skill with which they are applied in the evaluation. Task Orders are utilized by Districts, Divisions, and research and development organizations.

41. The contracting methods discussed in this section can all be used for contracting biological and chemical evaluations of dredged material. However, it is recommended that each CE District employ the contract method that best meets its particular need or preference.

Contract Management

42. Conscientious contract management is essential if satisfactory results are to be achieved. Poor management can negate or invalidate the entire study. Since contract management is so vital to the success of a project, it is appropriate that contract management be discussed.

Quality control and quality assurance

43. The terms quality assurance (QA) and quality control (QC) are often used synonymously, although they represent two distinct concepts. In broad terms, QA is the overall program that specifies the QC practices applied to the many individual aspects of a program.

44. More specifically, QC includes those actions taken by laboratories in day-to-day activities to achieve desired precision, accuracy, reliability, and comparability in the results obtained from sampling and analysis activities. Quality assurance includes those actions taken to ensure that laboratory QC policies and procedures are being properly implemented.

45. Engineer Regulation 1110-1-8100 defines contractor QC and Government QA as follows:

a. Contractor quality control is the testing and inspections performed for or by a contractor in controlling his procedures, laboratory equipment, and personnel so that the completed function will comply with the contract requirements.

b. Government quality assurance is the testing, evaluations, and inspections performed by the Government in verifying that (1) the contractor conducts the project in compliance with the contract requirements, (2) the end results meet contract criteria, when furnished by the appropriate Corps office, and (3) the contractor's laboratory equipment, personnel, and procedures are adequate for quality control.

46. Quality assurance must be a prime factor in project planning, scope-of-work preparation, contractor evaluation and selection, and management of a project. It is only in this manner that a project manager can be reasonably sure of providing a quality product.

47. A quality assurance plan must be designed as a management tool that can be implemented within the limits set by procurement policy. The plan must also provide minimum qualification and performance standards from which scopes of work can be written and potential contractors evaluated. The preaward and postaward quality assurance required of potential contractors must be clearly understood as technical performance specifications. Postaward contract management must clearly define data verification procedures and minimum acceptable performance and quality control activities that will be conducted and documented.

48. If subcontractors are used for either sampling or analysis, the prime contractor should have full responsibility for the quality assurance of the subcontractors' efforts. The subcontractor's role in the project (sampling and analyses) should be thoroughly identified in the technical proposal or bid. The quality control program of the subcontractor must be included in the technical proposal or bid and should meet the same requirements that would be expected of the prime contractor if the entire job were being conducted in-house. Reporting practices and lines of authority between contractor and subcontractors should be closely scrutinized to ensure that prompt action can be taken if quality control problems develop with the subcontractors. Written reports should be required between the contractor and subcontractors if quality control problems arise.

49. General quality assurance considerations applicable to both biological and chemical evaluations are addressed in Part III. Quality control and quality assurance for biological and chemical evaluations will be addressed in detail in Parts IV and V, respectively.

Progress monitoring and report review

50. The contractor is responsible for the timely and satisfactory performance of his contract. However, the Government cannot rely entirely on the contractor to make sure that the contract work is progressing as scheduled; it cannot risk poor performance. These failures may cause costly delays in the contract or the program of which it is a part. Thus, the Government monitors contract performance closely to ensure that desired end items are delivered on

time. The method of monitoring that is used depends on the length, complexity, and urgency of the contract.

51. Monitoring serves many purposes. It may provide up-to-date information. It may help to isolate performance problems. It may help to determine future funding requirements by comparing actual costs with progress. Monitoring can help in determining the Government's rights under the contract. It may also help in determining the adequacy of the contractor's own monitoring system. In each case, the purpose of monitoring is to obtain the information the Government needs about the particular procurement.

52. The relatively short time period usually required for dredged material evaluation contracts will normally limit monitoring to informal contacts, usually by telephone or by occasional laboratory visits. However, these contacts are very important in that they convey the importance of the work to the contractor and convey the Government's interest in completing the work on schedule. Specifically, these contacts can inform the project manager when the analyses have begun, how they are proceeding once under way, and when they are completed. The contacts can also inform the project manager of the status of subsequent data reports. Summaries of all telephone calls should be distributed to appropriate CE personnel. Should the project manager detect a potential problem through a telephone call or visit, he/she can alert procurement personnel and the Contracting Officer. This can possibly be followed with an official letter of concern to the contractor, reminding him of his contractual obligations.

53. The contract should stipulate that the contractor submit draft and final data reports as well as monthly progress reports on the status of the study, including QA/QC results. The draft report should be written at the completion of all analyses. Upon receiving the draft report, the project manager should distribute copies to appropriate personnel for simultaneous review. All comments should then be collected and reviewed by the project manager with relevant comments being presented to the contractor. After the contractor has been provided sufficient time to study Corps comments, and depending on the nature of the comments, a meeting should be convened, or a telephone conference call held, between Corps and contractor personnel to discuss resolution of the comments. Finally, the contractor should make the directed changes to the draft report and submit the revised report, as a final report, to the agency for acceptance.

Contract closing and payment

54. The project manager should request the contractor to bill the agency if it is not done promptly upon acceptance of the final report. The project manager should also request finance personnel to pay the contractor if payment does not occur promptly after the final report is accepted and the bill is received. These steps will help establish and/or maintain Corps-contractor rapport.

Classification of Reports

55. Proper QA/QC guidelines and contract management will ensure that data collected and contained in the draft contract report will be of the highest quality and can be used for the intended purpose. Evaluation and/or assessment of the scientific data will be used as an integral part of the decision-making process by the Corps and the USEPA to ascertain the suitability of dredged material for disposal.

56. If evaluation of the data shows that potential ecological harm does exist from sediment disposal, the appropriate management strategy for disposal of dredged material must be instituted. This will include consideration of disposal alternatives and steps required for selection and implementation of appropriate disposal management strategies.* However, if the evaluation of data shows that disposal will not adversely effect the environment, conventional open-water or confined disposal without restrictions would be appropriate. This information should be made available for intraagency, interagency, or public use.

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* Francingues, N. R., Jr., Palermo, M. R., Lee, C. R., and Peddicord, R. K. 1985. "Management Strategy for Disposal of Dredged Material: Contaminant Testing and Controls," Miscellaneous Paper D-85-1, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

PART III: QUALITY ASSURANCE CONSIDERATIONS APPLICABLE TO BOTH BIOLOGICAL AND CHEMICAL PROCEDURES

Project Planning

57. Quality assurance and quality control planning is necessary to ensure that the biological and chemical data generated meet project needs. The Request for Proposals or Invitation for Bids should be as detailed as possible without restricting innovation. Most environmental data are collected in response to regulatory investigations; therefore, it is essential that the analytical process meet regulatory criteria. The CE should require the contractor to provide a project QA/QC plan similar to the one that the US Environmental Protection Agency requires all of its contractors and grantees to prepare. The elements of this plan are given in below. Details on each element are provided in USEPA (1986).

- Title page
- Table of contents
- Project description
- Project organization and responsibilities
- QA objectives
- Sampling procedures
- Sample custody
- Calibration procedures and frequency
- Analytical procedures
- Data reduction, validation, and reporting
- Internal quality control checks
- Performance and system audits
- Preventive maintenance
- Specific routine procedures used to assess data precision, accuracy and completeness
- Corrective action
- Quality assurance reports to management

Field Activities

58. Field quality assurance should be an integral part of the sampling plan and should begin with the design of the plan to ensure that sampling efforts will accomplish the desired objectives. Since the field quality control measures are inherently more difficult to monitor, it is essential that detailed sampling plans be written and reviewed prior to initiation of sample collection. Among the reviewers should be (a) an experienced member of the field collection team who can offer suggestions based on actual field

involvements; (b) an analytical chemist familiar with requirements for sampling containers, preservation, holding times, and volumes, who can also review the applicability of standard operating procedures, ensure the collection of quality control samples such as blanks, spikes, duplicates, and split samples that are necessary to control the sampling process, and ensure that documentation is specified; (c) an engineer or scientist familiar with the overall objectives of the project to ensure that samples are representative and that variations in the sampling process have been considered, (d) a statistician to verify that the sampling approach will be adequate for any statistical calculations or decisions, and (e) a safety expert to consider any special requirements from a safety perspective.

59. The sampling component of the QA plan shall include the following documentation: (a) accepted sampling techniques, (b) prefield activities such as equipment check-out, calibrations, and container storage and preparation, (c) field QC procedures, (d) information to be included in field logbooks and sample labels, (e) post-field activities, including sample shipment and receipt, chain-of-custody procedures, and equipment check-in, and (6) generation of QC samples and use to be made of QC samples.

60. A chain-of-custody procedure is written documentation used to trace sample possession from collection to final disposal and provides credibility to the analytical process if litigation is involved. A detailed chain-of-custody procedure applicable to water, sediment, or tissue samples is presented in Appendix B.

61. Training the field crew is another aspect of QA/QC and is an essential part of a successful sampling operation. The field crew must be familiar with all terms used in the sampling plan and must be cognizant of the need to collect samples properly to avoid cross-contamination. Qualifications for the field supervisor should include a bachelor's degree in Chemical, Physical, Biological, Environmental Science or Engineering, with 2 or more years of field or laboratory experience.

62. The field logbook kept by the field supervisor must be a detailed account of significant field activities and observations relevant to sample quality. The logbook should contain enough information that the sampling process can be reconstructed without reliance on the sampler's memory. As a minimum, the logbook must contain the following information:

- Project name
- Name and address of field supervisor
- Sample site location
- Observations at sampling site
- Date and time of collection
- Sampling methodology
- Type of sample (water, sediment, tissue, etc.)
- Type of sample (water, sediment, tissue, etc.)
- Number of samples taken
- Field sample identification
- Identity of sample collectors
- Preservation
- Method of packaging and shipping or transporting
- Any field measurements recorded
- Notation whether chain-of-custody procedures are in effect

63. Plumb (1981) discusses selection of sampling locations within a dredging site and offers a protocol for balancing project and economic objectives in determining the number of samples to be collected. The project manager should calculate at least 10 percent additional cost for quality assurance analyses. The techniques presented in Appendix C can be useful in reducing the cost of sediment characterization. Plumb (1981) and Rochon and Chevalier (1987) provide good discussions on sample procedure selection, limitations and merits of various water and sediment samplers, sample handling, and preservation techniques.

64. Samples should be shipped or transferred to the laboratory as soon as possible to ensure that the integrity of the samples is maintained. In addition, the field supervisor should inspect the shipping containers to confirm that samples are sufficiently iced to maintain the temperature at 2° to 4° C.

65. Appendix D outlines techniques for collection, preservation, and storage of dredged material samples in compliance with the Ocean Dumping Act (Battelle 1988).

Preaward Inspection and Testing

66. Preaward inspection of offeror laboratories provides an excellent way to assess their QA program, analytical facilities, and bioassay facilities. Such inspections should be conducted as early in the contractor evaluation process as practical, since they can often provide very good insight into the contractor's real experience and capability to perform the

work properly and in a timely manner. All communications with the offerors must be conducted in accordance with Corps contracting procedure. It is therefore mandatory that the project manager contact the contract and/or legal branches at the District prior to initiating any preaward laboratory inspections.

67. Criteria for deciding if a preaward survey is needed are presented in Table 6 of a USEPA report by Stratton and Bonds (1979). Stratton and Bonds (1979) also give a suggested preaward inspection agenda for QA aspects of sampling and chemical analysis. Guidance can also be found in the Corps Engineer Technical Letter (ETL) 1110-2-309, Water and Wastewater Laboratory Inspections, dated 5 February 1988. Assessing the bioassay capabilities of an offeror during a preaward inspection is discussed in Section 4.01C of this ETL.

68. Performance test samples are also useful in evaluating a potential contractor. Performance samples may consist of water, tissue, or sediment. In the past, these samples usually consisted of those available from the National Institute of Standards and Testing, formerly the National Bureau of Standards. However, since most reputable laboratories today routinely use the NIST reference materials, other sources such as Environment Canada or other commercial sources should be investigated for use.

69. If the CE Project Manager is uncertain about the need for performance test samples, the option of requiring them should be stated in the solicitation. The following paragraph is suggested (Stratton and Bonds 1979):

> Offerors whose proposals are determined to be technically acceptable under the initial evaluation criteria stated herein may be required to demonstrate acceptable performance by analyzing not more than _____ unknown samples for _____ parameters per sample. The cost for analysis of these samples shall be at the offeror's expense. Sample analysis results shall be compared to known results for scoring purposes.

70. Stratton and Bonds (1979) suggest using performance test samples when the work involved will exceed $25,000 to $50,000 in cost, when analytical QA is very important to the success of the project, when there is doubt about the performance of offerors, or when offeror(s) cannot provide documented results of previous performance tests. The number of performance test samples should be limited due to time and cost requirements placed on the offerors.

Data Examination

71. An important aspect of quality assurance is the proper and accurate reporting of data. This depends primarily upon careful data handling by the contractor. However, CE personnel can minimize difficulties by reviewing data for apparent inconsistencies at the earliest opportunity (e.g., progress letters, draft reports, etc.). All chemical results, including bioaccumulation data, must be specified as being on either wet or dry weight basis, and units must be given. Replicate bioaccumulation values for a single species and contaminant which differ from their mean by more than two standard deviations should be verified for correctness. Decimal point slippages are one possible cause of such situations. A statistician should be consulted for more detailed advice on dealing with apparently anomalous data points.

72. Analytical errors and incorrect reporting of data can be corrected, but both are easier to identify and to correct early in a project than if they are not detected until the final report. For example, if survival is reasonably consistent among four replicate bioassay containers, but markedly lower in the fifth, conditions during the test should be carefully examined. If an obvious and reasonable explanation is found (e.g., reduced water flow to that aquarium, loss of aeration in that container), this should be explained in the report and kept in mind when interpreting the data. Inconsistencies in the data often are due to errors which are easily correctable; however, if no errors are found, the data must be regarded as an acceptable indication of the true response of the organisms.

References

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Plumb, R. H., Jr. 1981. "Procedures for Handling and Chemical Analysis of Sediment and Water Samples," Report EPA/CE-81-1, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

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Stratton, C. L., and Bonds, J. D. 1979. "Quality Assurance Guidelines for IERL-CI Project Officers," EPA 600/9-79-046, Industrial Environmental Research Laboratory, US Environmental Protection Agency, Cincinnati, OH.

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PART IV: LABORATORY SELECTION AND CONTRACT MANAGEMENT FOR DREDGED MATERIAL BIOASSAY AND BIOACCUMULATION STUDIES

General

Importance of reliable bioassay data

73. Section 404 of the Clean Water Act authorizes the CE to issue permits for the discharge of dredged or fill material into waters of the United States in accordance with 404(b)(1) Guidelines. Dredged material must be tested in order to determine the potential for unacceptable adverse ecological effect on the environment, unless it meets exclusions specified by 40 CFR 230.4-1(b)(1). Test data are used as an integral part of the decision-making process by the CE and USEPA regarding the suitability of dredged material for disposal. Therefore, it is vital that the test data submitted in support of private permit applications or Federal projects be of the highest quality.

74. The CE must use every means available to ensure that tests conducted can be repeated when necessary, yielding substantially the same results, and that the data are obtained in a competent fashion and can be defended against legal-scientific challenge. Use of QA techniques by private firms conducting dredged material bioassays and an awareness of QA by CE project managers will help ensure the quality of dredged material test data. Quality assurance techniques, when used properly, will help provide project managers with confidence that laboratory tests and procedures have produced data that are reliable and can be used as the basis for important regulatory decisions.

Biological testing

75. Biological testing is part of the national comprehensive tier testing approach (Table 1) supported by the CE (Engler et al. 1986). Each successive tier is based on a "reason to believe" that there is potential for unreasonable degradation of the aquatic environment. Each tier is optional and can be eliminated if sufficient information is available and provides no substantive reason to believe contaminants are present above reference site levels and that disposal may not cause unreasonable degradation of the environment. On the other hand, if historical information is inadequate or provides a substantive reason to believe contaminants are present above reference levels, testing is required. Since no single test can be applied in

Table 1

Comprehensive Testing Approach for Aquatic Disposal

As Part of the Federal Standard*

Tier I	Initial evaluation of existing information and "reason to believe there is contamination."	
Tier IIA	Bulk sediment inventory. Reason to believe dredged material is more contaminated than disposal site sediment and potential unacceptable adverse effects may occur.	
Tier IIB	Elutriate analysis. Chemical analysis for contaminant(s) of concern, contrast to appropriate water quality criteria and/or standard with consideration of mixing. Comparison to receiving water quality and/or bioassay when no standard exists.	
Tier III	Biological tests.	
Tier IIIA	Acute bioassay toxicity tests (as appropriate).	
	Water Column (Elutriate)	Select Species
	(Mixing considered)	(As necessary)
	Dissolved phase	Mysid shrimp
	Suspended solids phase	Grass shrimp
		Bivalve
		Fish
		Larva, bivalve
		Other
	Benthic	
	Solid phase	Mysid shrimp
		Amphipod
		Grass shrimp
		Clam
		Polychaete
		Other
Tier IIIB	Bioaccumulation.	
	Water Column	Select Species
	Suspended solids phase	Grass shrimp
		Clam
		Polychaete
		Other
	Benthic	
	Solid phase	Clam
		Polychaete
		Other

* This table presents the general types of tests and evaluations in a tiered and sequential basis where each tier (step) is optional and may be eliminated or chosen as appropriate. Species tested are not mandatory but represent those that can be considered in evaluating a proposed disposal site region (taken from Engler et al. 1986).

all cases to evaluate the effects of proposed discharges of dredged or fill material, 40 CFR 230.4-1 allows for multiple tests. The suitability of the proposed disposal sites may be evaluated by the use, where appropriate, of sediment analysis.

76. The first tier (Table 1) of the existing approach consists of an initial evaluation of historical data to establish whether there is a "reason to believe" that contaminants are present or not present. This tier is often referred to as the "exclusion clause" [40 CFR 230.4-1(b)(1)]. If the dredged material is not considered chemically contaminated and is physically/chemically similar to the substrate at the disposal site, no further testing is required. However, if there is a reason to believe that contaminants are present, or there is insufficient information, a second tier of evaluation may be conducted which consists of chemical inventory of the sediment. Should sufficient information be available from previous testing and evaluation, no additional chemical analyses are necessary.

77. It has been shown by Lee and Plumb (1974) that bulk sediment analysis was not adequate to assess the effect of contaminants on water quality. In addition, the chemical composition of the dredged material alone is not an effective indicator of the bioavailability of the contaminants. DiSalvo and Hirsch (1978) showed no relationship between bulk sediment analysis and bioaccumulation by aquatic organisms. However, bulk sediment analysis is a viable tool to inventory contaminants of concern and compare the chemical composition of the dredged material to the composition of the material at the disposal site, focusing on contaminants of concern, such as heavy metals, polychlorinated biphenyls (PCBs), polynuclear aromatic hydrocarbons (PAHs), pesticides, oil and grease, and other substances of ecological or human concern. If contaminant concentrations in the dredged material are significantly greater than those observed in the dredged material at the disposal site and there is reason to believe that the substances are bioavailable, a third tier of testing may be required.

78. The standard elutriate test (USEPA/USACE 1977) is appropriate for evaluating the potential for dredged material disposal to impact the water column. Since this test includes contaminants in both the interstitial water and the loosely bound (easily exchangeable) fraction in the sediment, it approximates the fraction of chemical constituents that is potentially available for release to the water column when sediments are dredged or disposed.

The results of the elutriate test are compared to water quality standards after consideration of mixing as described in the 404(b)(1) Guidelines. If there are no water quality standards or the standards are thought to be inadequate, a suspended particulate phase bioassay may be conducted along with consideration of mixing.

79. If there is concern regarding impacts to benthic organisms, a benthic bioassay may be conducted. The benthic bioassay is a comprehensive assessment used to ascertain the potential effect of contaminated dredged material on aquatic biota. This is done by using three aquatic organisms: a filter-feeder, a deposit-feeder, and an infaunal (burrowing) organism. These organisms represent different ecological niches at the disposal site, thereby providing a measure of biological impact for a wide range of assimilative mechanisms to which the contaminated dredged material might be exposed.

80. If there is reason to believe that bioaccumulation is of concern, a second component of the third tier consists of evaluating the potential uptake of contaminants. This may involve utilizing the suspended solids and solid phases. The suspended solids phase evaluates the biological impact ascribed to the physical presence of suspended particles and any biologically active contaminants associated with the particulate or dissolved fraction. The major evaluative efforts should be placed on the solid phase (USEPA/USACE 1977). It is generally felt that if a dredged material is going to have an environmental impact, the greatest potential usually lies in the solid phase because it is not mixed and dispersed as rapidly or to such an extent as the liquid and suspended particulate phases. Bottom-dwelling animals also live and feed in and on the deposited solid phase for extended periods.

81. Testing of the appropriate phase is determined by the reason to believe that a potential for unacceptable adverse impacts in one or more phases could occur. Flexibility is incorporated in the approach in relation to the selection of bioassay species to be used in the tests. The intent is to evaluate the potential impact on a deposit-feeder, a burrower, and a filter-feeder representative of major ecological compartments (Engler et al. 1986). The organisms used in bioassays must be appropriate and most sensitive (40 CFR 227.27, paragraphs c and d) to evaluate the potential impact of contaminated sediment on the aquatic biota. Tables 2 and 3 (based on USEPA/USACE 1977) give general categories of organisms that are considered appropriate and sensitive.

Table 2

Recommended Appropriate Sensitive Marine Organisms for Use in Liquid Phase and Suspended Particulate Phase Bioassays*

Zooplankton	Crustacean or Mollusc	Fish
Copepods, *Acartia* sp.	Mysid shrimp, *Mysidopsis* sp.** *Neomysis* sp.**	Group I
Larvae of recommended crustacean or mollusc species	Grass shrimp, *Palaemonetes* sp. *Palaemon* sp.	Silversides, *Menidia* sp.
	Commercial shrimp, *Penaeus* sp.	Pinfish, *Lagodon rhombiodes*
	Sand shrimp, *Crangon* sp.	Spot, *Leiostomus xanthurus*
	Oceanic shrimp, *Pandalus* sp.	Shiner perch, *Cymatogaster aggregata*
	American lobster, *Homarus americanus*	Group II
	Blue crab, *Callinectes sapidus*	English sole, *Parophrys vetulus*
	Cancer crab, *Cancer* sp.	Flounder, *Platichthys* sp. *Paralichthys* sp. *Limanda* sp.
	Amphipods, *Ampelisca* sp. *Paraphoxus* sp.	Group III
	Cumacean, *Diastylopis* sp.	Sheepshead minnow, *Cyprinodon variegatus*
	Macoma clam, *Macoma* sp.	Mummichog, *Fundulus heteroclitus*
	Nucula clam, *Nucula* sp.	Killifish, *Fundulus* sp.
	Yoldia clam, *Yoldia* sp.	
	Surf clam, *Spisula solidissima*	
	Hard clam (quahog), *Mercenaria* sp.	
	Ocean quahog, *Arctica islandica*	
	Scallop, *Argopectin* sp. *Aequipectin* sp.	
	Gemma clam, *Gemma gemma*	
	Edible mussel, *Mytilus edulis*	

* Lists are not in order of preference or desirability except for the groups of fish.
** All liquid phase and suspended particulate phase bioassays should include one of these species.

Table 3

Recommended Appropriate Sensitive Benthic Marine Organisms for Use in Solid Phase Bioassays*

Crustacean	Infaunal Bivalve	Infaunal Polychaete
Mysid shrimp, *Mysidopsis* sp.** (D) *Neomysis* sp.** (D)	Macoma clam, *Macoma* sp. (F, D)	*Neanthes* sp. (D, B)
Infaunal amphipods,	Nucula clam, *Nucula* sp. (F, D)	*Nereis* sp. (B)
Ampelisca sp. (F, D) *Paraphoxus* sp. (F, D, B)	Surf clam, *Spisula solidissima* (F)	*Nepthys* sp. (B)
Grass shrimp, *Palaemonetes* sp. (D) *Palaemon* sp. (D)	Hard clam (quahog), *Mercenaria* sp. (F)	*Glycera* sp. (B, D)
Commercial shrimp, *Panaeus* sp. (D)	Ocean quahog, *Arctica islandica* (F) Gemma clam, *Gemmà gemma* (F)	*Urechis* sp. (B, F) *Magelona* sp. (B, D)
Sand shrimp, *Crangon* sp. (D)	Littleneck clam, *Prototheca staminea* (F)	*Owenia* sp. (B, D)
Oceanic shrimp, *Randalus* sp. (D)	Cockle, *Clinocardium nuttali* (F)	*Diopatra* sp. (B, D)
Blue crab, *Callinectes sapidus* (D)		*Glycinde* sp. (B)
Cancer crab, *Cancer* sp.(D)		
Cumacean, *Diastylopsis* sp. (F, D, B) *Diastylis* sp. (F, D, B) *Lamprops* sp. (F, D, B)		

Note: Parenthetical notations as follows: F - filter-feeding species, D - deposit-feeding species, and B - burrowing species.

* Lists are not in order of preference or desirability.

** All solid phase bioassays should include one of these species.

82. The WES report entitled "Considerations in Selecting Bioassay Organisms for Determining the Potential Environmental Impact of Dredged Material" (Shuba, Petrocelli, and Bentley 1981) will be useful to CE project managers in the selection of test species. The experiences of personnel from CE Districts throughout the coastal United States in choosing appropriate bioassay organisms are discussed. A list of considerations that could be used to help rank potential test species was developed (Shuba, Petrocelli, and Bentley 1981):

a. The species, or a closely related one, is found at the disposal site.

b. The species is readily available through field collecting or purchasing.

c. A toxicological data base, preferably one including responses to dredged material, exists for the species.

d. Response to the same toxicant is reproducible.

e. The species can be maintained in a healthy condition in the laboratory.

f. The species can be cultured and will reproduce under laboratory conditions, providing sensitive life stages and sizes for testing.

g. The species can be used in the major types of dredged material bioassays.

h. The species occurs over a wide geographic area.

i. The species is economically important.

j. The species is ecologically important.

k. The species is compatible with other test species.

83. Standard Methods (American Public Health Association (APHA) 1985) is also a compendium for selecting test organisms. This reference discusses the sensitivity of certain organisms to potential contaminants. It also gives a list of considerations that must be taken into account in selecting appropriate bioassay organisms.

Quality assurance and
dredged material bioassays

84. The CE guidelines for dredged material bioassays can help ensure the precision and accuracy of data by discussing proper reference and control sediments, requiring laboratories to document QA practices as recommended by various USEPA manuals, and subjecting laboratories to periodic inspections. These procedures will help achieve good QA once a contract is under way, but

do not address the important issues of initial selection of a good bioassay laboratory.

85. The CE can help ensure quality bioassay work by choosing a contractor that has some experience in the area of dredged material assessment or by conducting a thorough onsite visit to a potential laboratory. If laboratories without experience with dredged material bioassays respond to solicitations, it is necessary that knowledgeable CE personnel visit the laboratories.

86. During a preaward inspection, CE personnel should carefully examine the facilities to be used for the work (see paragraphs 94-113). The CE personnel should ask to see data sheets, equipment calibration logs, culture histories, and other indications of careful and thorough record-keeping. Determination of the need for a preaward inspection and likely items of concern are discussed by Stratton and Bonds (1979).

87. A laboratory with experience in dredged material bioassays would be expected to discuss that experience in its proposal in relation to the planned project and to list or provide copies of any reports or publications that would demonstrate expertise. In general, higher quality bioassays could be expected from a laboratory that:

a. Has been conducting aquatic, preferably sediment, bioassays for several years.

b. Has personnel with experience and published reports in bioassay-bioaccumulation work.

c. Maintains many of the test organisms in their facility during most of the year.

d. Has some experience with dredged material or sediment bioassay work.

e. Maintains written records of all previous tests with regard to conditions, calibration of equipment, etc., and has written standard operating procedures for bioassay tests available for inspection.

88. A laboratory without experience in conducting dredged material bioassays may be awarded the contract. In instances such as this, the laboratory should demonstrate familiarity with the literature on dredged material bioassays and with CE guidelines and have considerable experience with other kinds of aquatic bioassays. Any laboratory seeking dredged material bioassay work should have all facilities necessary for holding and testing aquatic organisms; these should be available for inspection at any time.

89. Some of the more important tasks that can be accomplished on a visit to a potential bioassay laboratory include observing the facilities that will be used for the work, talking with the scientists and technicians that will be involved, and observing any test organisms that are on hand. The project manager should know where the animals were obtained, how long they have been in the laboratory, and what system is used to provide them with high quality water. A laboratory that has all of the desired test species on hand and in good health should be viewed more favorably than one that does not have this capability. After a preaward visit to a laboratory, a brief document should be prepared identifying the laboratory by name and location, describing the bioassay and chemical equipment, identifying persons in charge, listing the organisms that are either on hand or have been tested successfully by the laboratory, and assessing the capability of the laboratory to provide high-quality results in a timely fashion.

90. Quality assurance of bioassay data differs in many ways from QA of analytical chemistry data due to the complex variables inherent in experimental work with living organisms. Factors that influence bioassay results include the overall health of the test organisms and individual organism variability, test temperature, salinity or hardness, water quality, and variability of test sediments. There is general agreement among aquatic toxicology researchers that sediment toxicity tests are more difficult to reproduce than the classic bioassay, in which fish are exposed to a well-defined constant concentration of a single toxicant dissolved in water. Sediment samples from the "same" location may vary greatly in the levels of contaminants present in geochemical forms that are biologically available. In bioaccumulation tests all of the above are important in relation to QA of the animal exposure conditions, and in addition there must be QA of the analytical procedures used to quantify the concentrations of contaminants in the animal tissues. The mysid shrimp may be considered, and has been widely used as an internal standard and basis for quality assurance.

91. In summary, the work involved in assessment of the toxicity and bioaccumulation potential of a number of sediment samples is complex and involves many areas where scientific judgment of the contractor must be relied upon. The IFB process makes it difficult to evaluate the scientific judgment of a potential contractor, which is an advantage of the RFP method. The process of choosing the most qualified bioassay contractor will be facilitated

as the CE acquires further knowledge of the available literature on QA and biological research and gains experience with various laboratories. This will also help ensure that quality data are obtained and that the work of awarding and managing a biological contract will be more efficiently accomplished in the future.

92. Appendix E contains additional references that are germane to biological testing. The list of references is not intended to be all-inclusive, but gives basic information that will enable the CE project manager to become familiar with biological testing and, ultimately, to increase the quality of biological data.

93. The following sections outline criteria for use in selecting a biological contractor. These include requirements with regard to facilities, test equipment, and testing procedures.

Physical Facilities

General

94. The Corps project manager must be familiar with the special laboratory requirements necessary for the conduct of reliable sediment bioassays. The reliability of a bioassay facility may be affected by the choice of materials used in the construction, the quality of construction, the consistency of operation, and the efficacy of the laboratory design in meeting the needs of test organisms and in achieving the required specifications of the bioassay. The following discussion is intended as general guidance, not as detailed and absolute requirements. It is entirely possible for laboratories not conforming to all details of this discussion to do very good work. However, laboratories that exhibit careful attention to detail and deal adequately with the topics addressed here are more likely to be consistently reliable.

Construction materials

95. All animal and sediment collection and transportation equipment and containers, culture vessels, holding tanks, test aquaria, aquarium fittings, and all components of water supply and temperature control systems must be made of noncontaminating materials. Aquaria may be constructed of glass, Plexiglas, epoxy-coated fiberglass, or fiberglass and epoxy-coated wood. Other materials suitable for use in pumps and piping and for water storage

include 316 stainless steel, titanium alloys, Teflon, polyvinyl chloride (PVC), linear polyethylene, nylon, polypropylene, silicone and vinyl tubing, and silicone sealant. Tygon and latex tubing have been shown to be toxic to marine phytoplankton, zooplankton, and bacteria; therefore, these should not be used in bioassays (Price et al. 1986). These authors also showed that pouring 200 ml of seawater through a 1-m piece of 0.6-cm-diam Tygon tubing removed the toxic effect, therefore allowing the use of Tygon tubing in bioassays. Copper or galvanized steel pipe and fittings, wood that has been painted, and rubber, with the exception of Buna-N, should not be used. Neoprene stoppers and surgical gloves are toxic to certain aquatic organisms, and these should not be used unless their acceptability has been demonstrated. Glass, silicone sealant, polyethylene, and polypropylene, while noncontaminating, may adsorb some metals and organic compounds such as PCBs and DDT, and by so doing may reduce the effects of a sediment being tested.

96. The contractor should specify all his materials in his response to a solicitation and be able to verify the nontoxic nature of his facility by demonstrating an ability to maintain viable cultures of bioassay test organisms, such as mysid shrimp, copepods, *Daphnia*, or algae. Although most organisms used in dredged material testing may be purchased or collected, these may be cultured in the laboratory. The presence of viable cultures kept over a number of generations demonstrates not only the nontoxic nature of the facilities but also a more general understanding of the needs of the organisms, and provides some assurance of the competence of the contractor.

Quality of construction

97. Good workmanship and design are two essentials for reliable and consistent performance of a bioassay laboratory. Faults in either of these areas will inevitably lead to breakdowns that can invalidate the results of a test. The Corps project manager should examine the bioassay facility carefully for the attention to detail that has gone into its design and assembly.

98. Permanent plumbing should be used whenever possible in the water delivery system; pipes and tubing should not be laid on the floor across walkways; there should be no leaks in the system as indicated by pooled water on the floor; and electrical equipment that might be subjected to water from any source should be protected with ground fault interrupter circuit breakers. Generally, the laboratory should be clean and orderly.

Holding and acclimation facilities

99. General. All holding and test conditions should be such that they meet the biological requirements of the test organisms. Proper holding and acclimation facilities will help reduce the frequency with which stressed organisms die. High mortality in the control would indicate the organisms were stressed from the start. The nonsediment-related stresses in addition to those attributed to the dredged material may be sufficient to cause higher exposed-group mortality than would have been the case with healthy animals. This situation would bias test results by embellishing the premise that the dredged material appears to be more toxic than actually the case. This potential problem can be alleviated by conducting bioassays with standard reference toxicants (Table 4). Standard reference toxicant bioassays provide evidence of the health of the test organisms and a general indication of the acceptability of laboratory techniques.

100. Standard toxicant bioassays are conducted by exposing organisms to a known toxicant using standardized conditions and techniques. Because of the uniformity of these tests, results with a given species can be expected to be consistently reproducible within an established range of variability. In order to be of relevance, however, these tests must be conducted with the same group of organisms as the companion dredged material bioassays and must use the same test conditions and techniques insofar as possible. The condition of the organisms used in every dredged material bioassay, as indicated by sensitivity to the standard toxicant, should be determined within the 7-day period preceding the bioassay. If preferred, this standard toxicant bioassay may be run concurrently with the dredged material bioassay, and laboratories conducting less than one toxicity test per month may find it convenient to do so.

101. Corps Districts have required standard toxicant bioassays to determine the 96-hr LC_{50} of sodium dodecyl sulfate for all species used in the liquid and suspended particulate phase bioassays (Krauser 1984). Such data should be compiled, and the overall mean 96-hr LC_{50} and its standard deviation should be determined for each species. This would provide an indication of the health of animals used in previous bioassays, against which the health of future test organisms, as measured by the sodium dodecyl LC_{50} values, could be compared. As an initial evaluative guide, standard toxicant LC_{50} values falling within ±2 standard deviations of the mean of previously determined values may be assumed to indicate that the companion dredged material

Table 4

Salient Features of Popular Reference Toxicants*

Reference Toxicant	Concentration Lethal to Various Organisms	Presumed Mode of Action	Comments
Sodium pentachlorophenate (NaPCP)	0.037 to 0.130 mg/ℓ 96-hr LC_{50} for underyearling rainbow trout, coho salmon, and sockeye; 3 mg/ℓ toxic to snail *Australorbis glabratus*	Uncouples oxidative phosphorylation, inhibits enzyme systems, and is considered to be a general metabolic stressor	Extensively used as a reference toxicant; an important industrial product; a wood preservative; biodegraded to trichlorophenols
Chromium (hexavalent)	100-hr LC_{50} for *Daphnia magna* 0.42 mg/ℓ; 96-hr LC_{50} values for three larval insects >17 mg/ℓ	Inhibits uptake of glucose in fish intestine	Used as a reference toxicant; actual concentration often considerably less than the calculated concentration
Dodecyl sodium sulfate (DSS) (also called sodium lauryl sulfate, SLS)	Fish and several estuarine crustaceans have 24-hr LC_{50} values between 1 and 10 mg/ℓ	Alters membrane permeability, gill damage to freshwater fishes and estuarine decapods, and damage to chemoreceptors	Required as a reference toxicant in oil-dispersant toxicity testing; high rate of biodegradation; used as a reference toxicant and considered to be rapid, nonselective, and consistent in toxicity
Sodium chloride	48-hr LC_{50} for *Daphnia pulex* 200 mg/ℓ; 96-hr LC_{50} for goldfish, 7,650 mg/ℓ; and for fathead minnow, 7,322 mg/ℓ	Induces osmoregulatory failure	Continuous-flow bioassay was not feasible due to the large amount required; not recognized as a toxic environmental contaminant
Phenol	48-hr LC_{50} was 21 mg/ℓ for *Daphnia magna*, 8.0 mg/ℓ for rainbow trout, and 36 mg/ℓ for fathead minnows	Causes paralysis in fish by increasing the quantity of acetylcholine released from the synaptic nerve endings	Used as a reference toxicant; toxicant loss in a period of several hours can be significant

* Taken from Laboratory Animal Management, "Marine Invertebrates," National Academy Press, 1981.

bioassays were conducted with acceptably healthy organisms. If the LC_{50} values fall outside this range, the sensitivity of the bioassay organisms, and therefore the overall credibility of the bioassay results, would be suspect. In such cases, the test facility and techniques should be examined for defects, and a different group of test organisms would be used in repeating the standard toxicant and dredged material bioassays. The extension of the standard toxicant testing to include organisms used in the solid phase bioassay may help resolve future concerns about toxicity and bioaccumulation data.

102. Although contractors will prefer to purchase some test organisms or collect them from the wild rather than culture them, the total aquarium space available for holding and acclimation should considerably exceed that which would be sufficient for conduct of the bioassay. Particular attention should be paid to these holding facilities because the care with which the test organisms are held and acclimated will determine their state of health at the start of a test, and this will have an important influence on their responses to test conditions. The attention the contractor gives to animal care is generally indicative of his overall attention to the details of good laboratory practice.

103. Static. Bioassay test organisms (i.e., fish, mysids, and shrimp) can be kept successfully in static systems using commercially available glass aquaria containing subgravel filters equipped with corner airlift apparatus. The appropriate medium, i.e. the "gravel" bottom, should be dolomite, crushed oyster shell, or coral. These will act as chemical buffers to keep the pH of the water stable. Quartz or silica sand should not be used as these have no buffering capacity. An external filter having activated charcoal under filter-wool is often used to supplement the subgravel filter, especially when the aquarium loading is fairly high. External filters will allow the organisms to be held for several days in aquaria.

104. When examining this type of holding tank, the Corps project manager should not be especially concerned about the presence of algae on the walls of the aquarium. Algae may be beneficial in that they will assist in the removal of a certain amount of nitrogenous wastes. However, it is important to note that algae respiration at night may significantly decrease dissolved oxygen. Removal of nitrogenous wastes, however, is primarily the task of the subgravel filter. The filter must be made "biological" by placing one

or two large "hardy" organisms such as lobsters, crabs, or fish in the aquarium not less than 2 weeks prior to use as a holding facility. The waste products from these organisms initiate the formation of a "biological" filter consisting of nitrifying bacteria that transform highly toxic waste ammonia to nitrite and finally to nitrate.

105. As long as an effective "biological" filter can be maintained, static systems work well. They require regular (once every other day) partial (~50 percent) water changes in order to dilute the constantly increasing concentrations of nitrate, sulfate, phosphate, and dissolved organics. Partial water replacements in static saltwater systems provide a convenient mechanism for maintaining stable salinity, which otherwise increases steadily due to evaporation.

106. Flow-through. If a flow-through capability exists, optimal holding conditions are possible; however, the advantages of a flow-through system are not realized without proper maintenance when standard rectangular glass aquaria are used. The sharp corners in a rectangular glass aquarium are unavoidable dead-spots where uneaten food and fecal debris will collect. Even small amounts of organic material in the water can stimulate the growth of disease-causing bacteria and parasites. This cannot be entirely avoided in rectangular aquaria even with a high flow rate. Therefore, the Corps project manager, in making his evaluation, should examine the corners of rectangular aquaria for organic material. The competent contractor will remove this material at frequent intervals. If standard 10-gal* glass aquaria are used in a flow-through solid phase bioassay, these considerations also apply to the test itself.

107. The best tank design for holding fish is a circular one with a circulating pump. These tanks are usually constructed of fiberglass with a removable central standpipe to carry away the overflow. Water is introduced into the tank at the side; the suction port for the circulating pump is located near the center of the tank and is screened. The discharge from the pump is through a 90-deg elbow plumbed into a bulkhead fitting on the side of the tank. This produces a circular current in the tank against which the fish can swim, and causes debris to be carried to the center of the tank where some

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* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page 4.

is lost in the overflow and the remainder is easily removed. Few testing laboratories are likely to have this type of holding tank.

108. Other less elaborate apparatus for holding fish and macroinvertebrates, such as the holding tanks described in Standard Methods (APHA 1985), will also give good service. The important point is not whether the tank is round or of another design, but that there are no sharp corners where organic debris will collect. If such a design is not used, diligent effort must be devoted to removing all debris from the tanks at frequent intervals.

109. Flow-through facilities are also desirable, but not essential, for culturing or holding mysids, shrimp, and other macrofauna. In the case of mysids, water must be flowing or moving constantly and consistently in the aquarium so that the organisms may orient themselves in the water column. If a static system is used to hold and culture mysids, this necessary water movement can be established by directing the air-lift tube from the undergravel filter downward and toward the center of the tank.

110. Bivalves and polychaetes may be kept in shallow plastic trays or on shallow wet tables under flowing seawater. If flowing water is not available, the organisms may be kept in glass aquaria equipped with air-lift corners or external charcoal and filter-wool filters. Bottom filters are not necessary or effective for keeping polychaetes or clams, as their burrowing activity continually disturbs the substrate and prevents establishment of a "biological" filter.

111. Most mussels do not require a sand substrate as they are not a burrowing organism. The holding facility requirements for mussels are similar to those of the polychaetes and clams.

112. The facilities for mass culture of zooplankton may be either static or flow-through. These are described in detail in both "Bioassay Procedures for the Ocean Disposal Permit Program" (USEPA 1978a) and Standard Methods (APHA 1985). The culture vessels are aspirator bottles or glass cylinders having a capacity of up to about 40 ℓ, equipped with a top-mounted low-rpm motor and stir-bar and having a source of cool-white fluorescent light.

113. During the holding and acclimation period (prior to testing), the contractor should keep detailed records on the aquaria that include the following: (a) date organisms were received; (b) temperature and salinity or hardness in which cultured or collected; (c) temperature and salinity or

hardness of laboratory receiving water; (d) pH, dissolved oxygen, temperature, and salinity or hardness to which organisms are acclimated; (e) original number of organisms; (f) mean weight and length of organisms, noted at the time of reception in the laboratory and at the start of a test; (g) mortality after collection and during pretest holding; and (h) water changing schedule.

Static Tests

Test containers

114. Static tests are dictated for liquid and suspended particulate phase bioassays by the volumes of test material that would be required for flow-through tests. Standard 10-gal all-glass aquaria may be suitable, but should not be rigidly required, as the volume and dimensions suitable for use in a bioassay are determined by a number of factors, not the least of which is the size of the individual organism. Liquid and suspended particulate bioassays are often conducted using fish, crustacean, and algal species. The 10-gal aquaria are suitable for bioassays involving fish (fry, juveniles, and subadults) but not for the much smaller zooplankton or algae. For these small organisms, other vessels are appropriate. Pyrex crystallizing dishes, 190 by 100 mm filled to a volume of 2 ℓ, have been used successfully in bioassays involving 20 mysids. An appropriate vessel for testing a similar number of copepods is a crystallizing dish 100 by 50 mm filled to a volume of 200 ml; 500-ml Erlenmeyer flasks are used for the algae.

115. Stackable culture dishes may also be used for the crustaceans, with the advantage that several of them may be placed vertically in a temperature and light-controlled environmental chamber, the bottom of the one above serving as a cover for the one below. If the dishes are not stacked, they should be provided with glass or Plexiglas covers. The advantage of low-profile, cylindrical vessels such as these over beakers or wide-mouthed jars is that they provide a large surface-to-volume ratio, permitting the crustaceans to distribute themselves optimally to minimize stressful crowding.

Temperature and photoperiod control

116. Temperature and light-controlled environmental chambers unquestionably provide the best conditions for the conduct of static bioassays using small containers. Commercial units are available which are capable of containing all of the crystallizing dishes or culture dishes necessary for a single zooplankton bioassay. Environmental chambers equipped with shaker

platforms are used for algal bioassays. The advantages of these chambers are that temperature can be controlled within the tolerances of the specified 20° ± 2° C; the desired light intensity, wavelength, and photoperiod can be provided; and the test can be conducted with a minimum of disturbance to the organisms. Walk-in environmental chambers having essentially the same features are also available or may be built to accommodate aquaria for conducting static bioassays with fish as well as with small crustaceans. The effect of photoperiod on sediment bioassays has received little attention. Laboratory tests are generally conducted under constant illuminations. These conditions tend to inhibit infaunal or epibenthic excursions. Most benthic invertebrates are exposed to subdued or no light in the environment. Consequently, conducting laboratory tests under constant illumination represents a worst-case scenario.

117. For fish and other large organisms, a workable alternative to the above facilities is the use of a temperature-controlled water bath. Water is circulated at a constant temperature around aquaria placed on supports in a bath. Temperature control can be achieved by the use of thermostatically controlled heater-chiller circulating units. The bath water must be kept free of algae to prevent a reduction in flow due to clogging and subsequent possible damage to the pump and heater units.

118. A facility using water baths should be located in a quiet, low-traffic area in the laboratory, and a constant ambient air temperature near 20° C should be maintained. Lighting is provided by suspending fluorescent fixtures above the baths and controlling the light-dark cycle with a timer.

119. The least acceptable arrangement for the conduct of static bioassays is the "table-top" approach in which reliance for temperature control is placed on maintaining ambient air temperature at a constant 20° C. This goal may be approached in a well-insulated building equipped with a highly efficient air conditioning system, but is otherwise unlikely. If a contract is let to a contractor who uses this approach, the contractor should be required to install a sensitive, continuously recording air temperature thermometer in the vicinity of the test chambers and to at least manually record water temperatures morning and evening in a simple-random sampling of the aquaria and test dishes. These data must be available to the Corps project manager.

120. Temperature should be maintained within a range of 4° C. This is certainly liberal, and any contractor should be capable of achieving this. Some added consideration should be given to a contractor who may have the proven ability to maintain water temperature control within stricter tolerances than this minimum.

Algal bioassay additional requirements

121. Algal bioassays are conducted to evaluate potential water column impacts. They require the use of sterile technique, and for this purpose an autoclave is essential. Algal growth behavior is highly dependent on lighting duration, wavelength, and intensity. Illumination should be by "cool-white" fluorescent lighting and should provide 4,304 lumens (400 ± 10 percent foot-candles) measured at the liquid level of the incubation flask (USEPA 1978b). An accurate light meter is required to obtain and verify the correct luminosity. This light meter must be standardized against a calibrated lamp obtained from the National Institute of Standards and Testing (NIST). Algal growth may be evaluated by using a spectrophotometer to measure optical density of a culture, or a fluorometer to measure chlorophyll a. Counting of algal cell density can be done using a compound microscope and a hemocytometer and ocular micrometer. The instrument of choice for counting algal cell density, however, is an electronic particle counter equipped with a mean cell volume computer. This instrument is relatively expensive, and special training is required to operate it properly. However, advantages in accuracy and in ability to rapidly count a large number of samples are strong points in favor of using an electronic particle counter for laboratories performing routine algal bioassays. Other equipment and supplies are listed in the USEPA manual "The *Selenastrum capricornutum* Printz Algal Assay Bottle Test" (USEPA 1978b), and testing should be conducted following the guidance given therein.

Flow-Through Test Aquaria

Aquaria

122. Standard 10-gal glass aquaria can be made suitable for flow-through solid phase bioassays by providing a means of introducing water at one end of the aquarium and an overflow at the other end. Improved circulation is obtained if the inflow of water is directed downward 2 in. below the level of

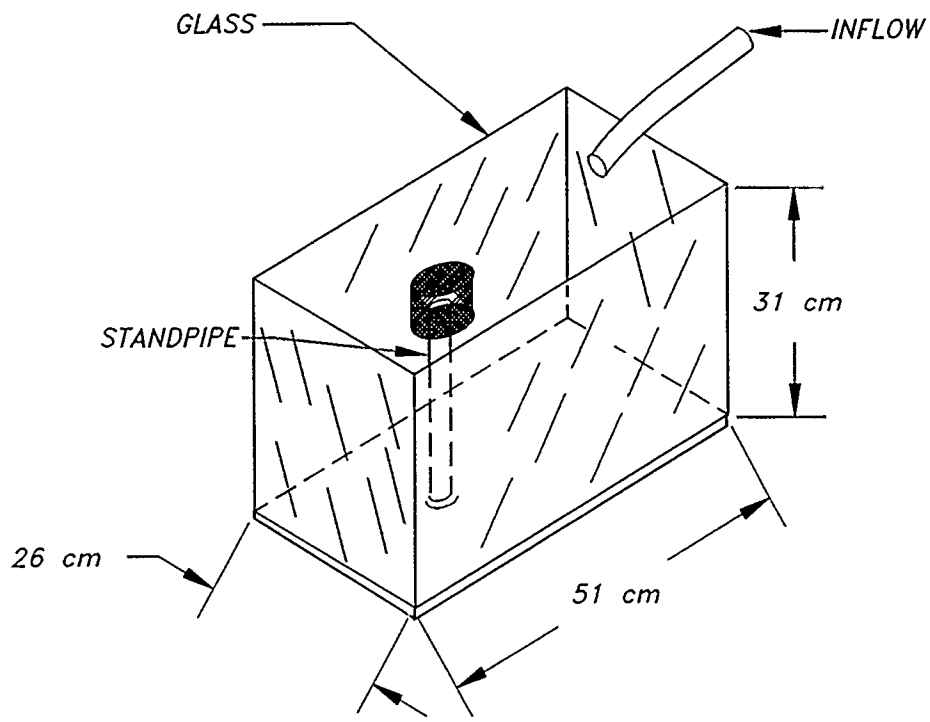
water at one end of the aquarium. Any means of accomplishing this consistent with good construction practices should be acceptable.

123. Overflow at the opposite end of the aquarium may be accomplished by installing either a screened standpipe in the aquarium bottom (Figure 1) or a screened port in the wall of the aquarium (Figure 2). A siphon that lifts water over the top of the aquarium must never be used. Such siphons are inherently prone to unpredictable failure with the result that water will overflow the tank.

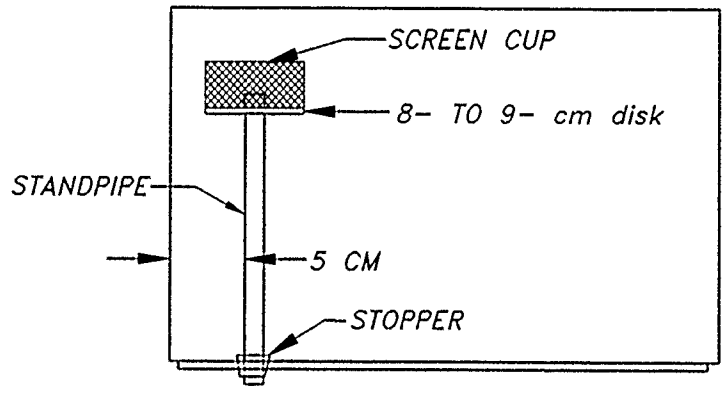
124. If the standpipe is used, a circular hole is cut in the bottom of the aquarium at the center of the end opposite to the inflow and about 5 cm from the inside wall (Figure 1a). The hole is of sufficient diameter to accommodate a stopper through which a 1/2-in. PVC pipe or similar-sized Plexiglas tube has been inserted. Alternatively, the hole is sized to accept a 1/2-in. PVC bulkhead fitting into which a 1/2-in. PVC pipe is threaded. The latter is a sturdier construction and is less prone to leakage. Around the top of the standpipe, an 8- to 9-cm-diam disk of Plexiglas or PVC is glued or threaded (Figure 1b). A cylinder of 0.5- to 1.0-mm screen, usually of some inert plastic material, is glued to the edge of the disk and rises above the waterline. The purpose of this design is to keep organisms away from the higher velocity area at the mouth of the overflow tube where they may be trapped and injured. Simply attaching a piece of Nytex or fiberglass window screen to the top of the overflow tube with a rubber band, for example, should be considered poor design. A variation on this design, which achieves the same purposes, should be considered acceptable.

125. An overflow may also be prepared by cutting a circular hole in the aquarium wall large enough to accommodate a stopper containing the overflow tube, or preferably a 1/2-in. PVC bulkhead fitting (Figure 2). The screen is of 0.5- to 1.0-mm plastic netting glued to a cup formed by an approximately 2-cm-deep by 8- to 10-cm-diam cylinder of Plexiglas or PVC, backed by a disk of the same diameter with a central orifice and connected to the stopper or bulkhead fitting by a suitable nipple.

126. On the outside of the aquarium, the wastewater should be carried away to a central manifold that is connected to a drain. When a water bath is used, it is poor design to simply allow the aquarium overflow to fall into the bath. In a solid phase sediment bioassay, it is inevitable that a certain amount of fine-grained solids will be suspended at some time during the test

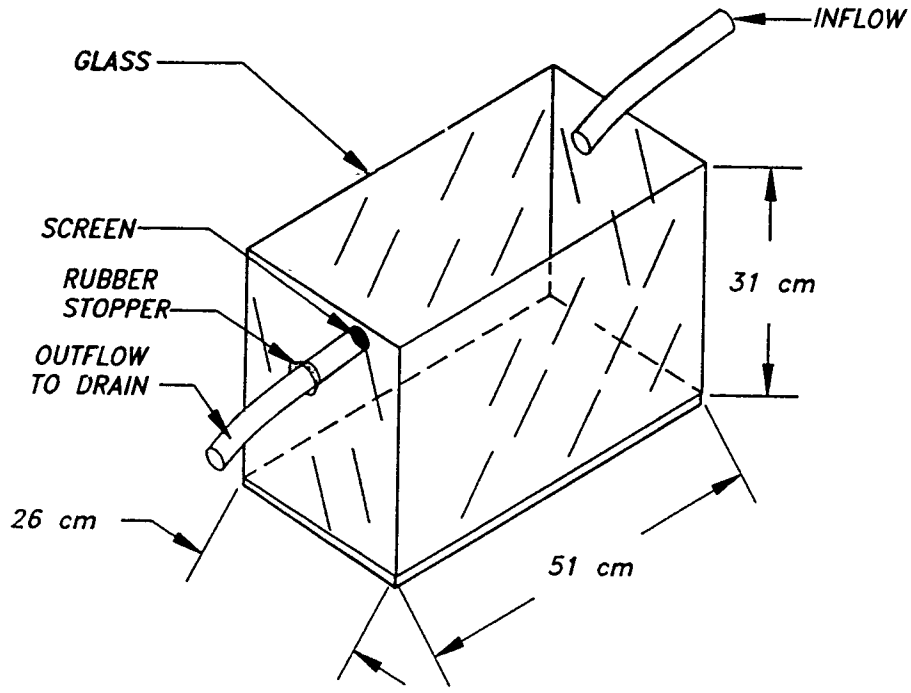


a. Screened standpipe

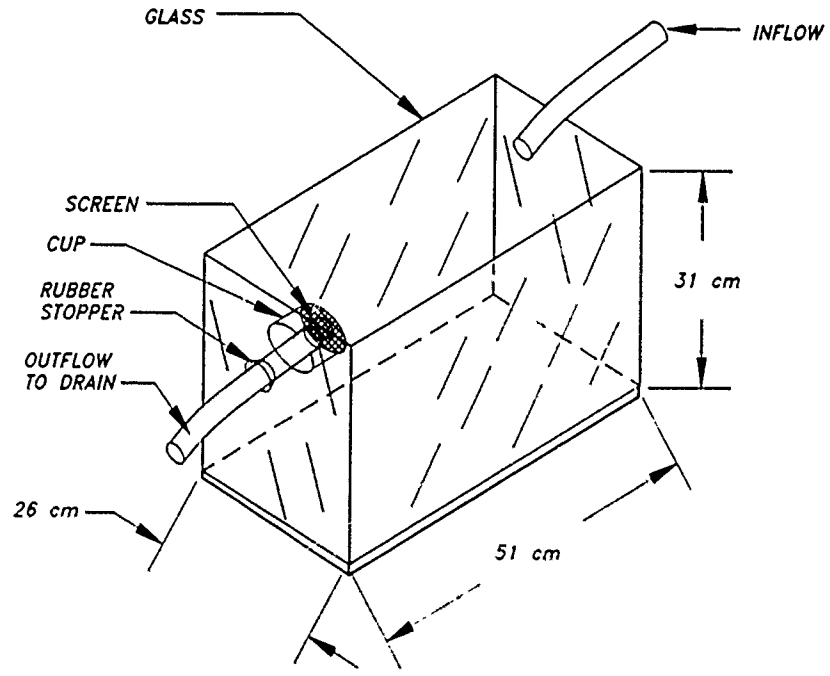


b. Details of standpipe

Figure 1. Flow-through test aquaria with screened standpipe



a. Screened port



b. Details of screened port

Figure 2. Flow-through test aquaria with screened port

and will be carried away in the overflow. If this material is allowed to mix with the water being used to maintain temperature in the bath, it will enter the circulating pump. Plastic pumps used in combined heater-chiller units are the most vulnerable to fine-grained abrasives, but the seals of most other pumps are also vulnerable. Properly designed water bath facilities will not mix waste with temperature control water.

127. The choice of either an overflow standpipe or an overflow port may be determined largely by the type of temperature control used. The simplest arrangement is to place the aquaria on runners set on a wet-table in a temperature-controlled room. The wet-table is usually shallow-sided and covered with fiberglass. It will have a large-capacity discharge at some point for carrying overflow from the aquaria to waste.

Temperature and photoperiod control

128. Temperature control of the aquarium water that depends entirely on control of room air temperature will likely meet the required specifications in this type of wet-table design only if the facility is set up in an actual environmental chamber. Water baths are generally the simplest and least expensive temperature control facilities that will provide the required accuracy.

129. As with the static bioassays, the flow-through facilities must be located in an area of low traffic flow and low noise. Photoperiod is controlled similarly using fluorescent light fixtures suspended over the aquaria and operated on a seasonally adjusted daylight to dusk schedule using a timer.

Flow Rate Control

130. A flow rate that will result in a 95-percent replacement time of approximately 12 hr (Sprague 1969) is the recommended replacement rate given in Standard Methods (APHA 1985) for bioassays involving water-dissolved toxicants. Standard Methods also cautions not to allow flow rate to deviate from this value by more than ±10 percent. This level of performance necessitates a relatively high degree of sophistication in the water delivery system.

131. Flow rate control has been a problem for workers in the field of aquatic toxicology for years, and the techniques for achieving good control are constantly being refined. A method that is used by some contractors involves manually adjusted valves or tubing pinch-cocks delivering water from

a constant-head tank by gravity flow. No assumptions should be made regarding the consistency of performance of such systems. At the low flow rate specified for these bioassays, it is very difficult to maintain flow within the suggested ±10-percent tolerance by this method.

132. If this method for water delivery is used, a contractor should be required to document the performance of his system by measuring flow rate into each test aquarium at least twice daily for an interval prior to and during a test, or until constancy of water delivery rate has been established, and periodically thereafter. Regardless of what method is used, flow rates to the individual aquaria should be measured frequently, and records should be kept.

133. A variety of methods can be used to obtain accurate and consistent flow rates. Variable-speed tubing pumps have been used, but these are expensive and require frequent attention to the condition of the tubing. Only Silastic tubing should be used, and it is expensive. Even Silastic eventually will collapse and can break, causing a complete disruption of flow rate unless it is pulled through the pump at the recommended intervals.

134. The apparatus introduced by Rubenstein et al. (1980) (Figure 3) provides a simple means of obtaining highly constant flow rates. The apparatus requires only one electric solenoid valve for each set of aquaria. A volume of water slightly in excess of that required to pass through the aquaria is introduced into a head box and flows into a double-celled chamber. The water overflows from the introduction chamber into the splitter chamber in which there is a drain standpipe slightly lower than the overflow wall. This allows a constant level of water to be maintained in the splitter chamber. In the center of the splitter chamber is a large stopper containing a number of equal-length small-bore standpipes equal to the number of aquaria to be served. The small-bore standpipes dump water into larger bore delivery tubes. The junction of the small-bore and large-bore tubes is open to the air. The rate of flow is regulated by adjusting the distance between the head of water and the top of the small-bore standpipes. Although few laboratories are likely to be using the system at present, it has proven reliable and is easily constructed.

135. A second type of apparatus that is accurate and reliable is a modification of the serial dilution apparatus originally introduced by Mount and Brungs (1967). In this apparatus (Figure 4), water flows into a Plexiglas trough divided into a number of equal-volume sections. The water is

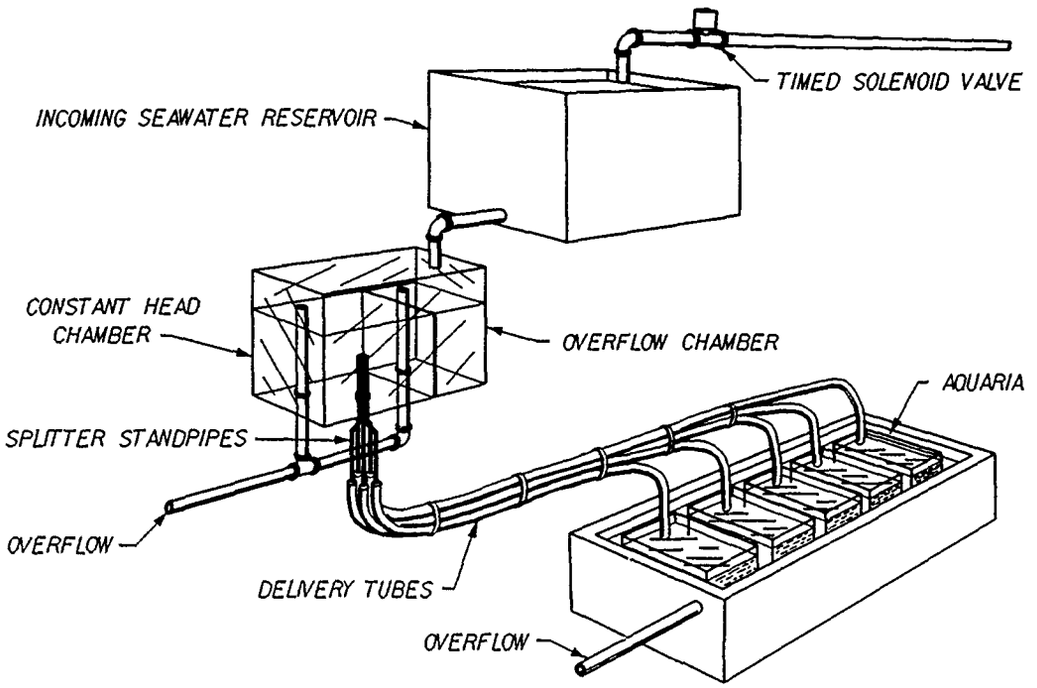


Figure 3. Constant-flow rate water delivery apparatus (after Rubenstein et al. 1980)

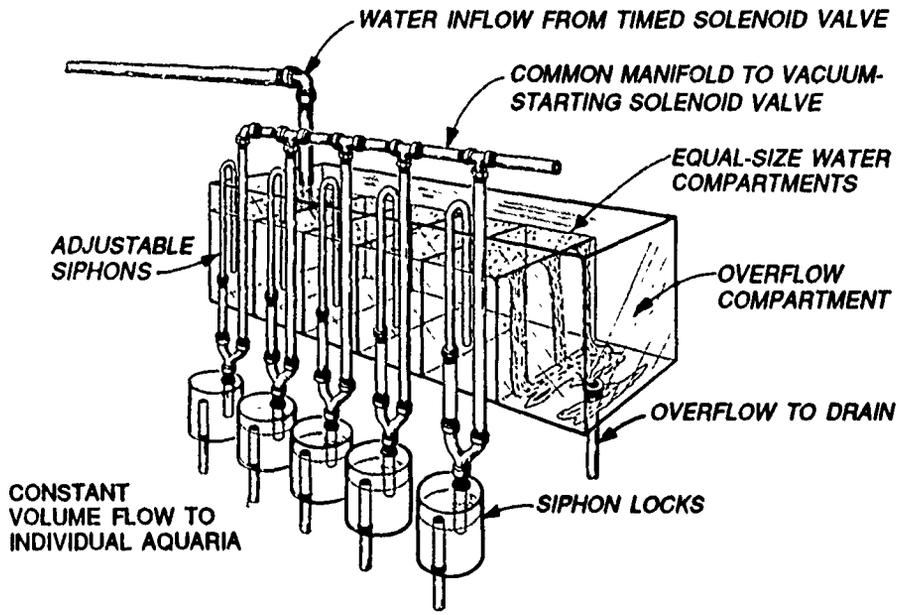


Figure 4. Dilution apparatus adapted to give constant-flow rate control for flow through sediment bioassay applications (after Mount and Brungs 1967)

introduced through an electric solenoid valve and flows until all sections are completely filled. The excess goes to drain, and the flow of water is stopped when all the sections are filled. Each section has an adjustable siphon running to a wye tube (resembles letter y in shape) inserted in a siphon lock. The other end of the wye tube is manifolded to a common suction line. The tube from the base of each siphon lock leads to an aquarium. Momentary activation of the suction starts the siphons, and water flows into the aquaria until the adjusted volume has been delivered and the siphon breaks. The cycle starts again after a timed interval.

136. Recent modifications of this design have improved on its performance and reduced its cost of construction (Garton 1980, Hemmer 1980). The apparatus is usually employed as a diluter in a toxicant delivery system for aquatic bioassays, but its efficacy in delivering water continuously at a highly controlled flow rate makes it suitable for use in flow-through dredged material bioassays.

137. A third apparatus that was developed by V. A. McFarland and C. H. Lutz of the US Army Engineer Waterways Experiment Station is the Flow-Through Aquatic Toxicology Exposure System (FATES). This system (Figure 5) is a unique and highly valuable research facility for creating carefully controlled suspended sediment load. It consists of 24 round-bottomed, cylindrical-shaped, 75-ℓ aquaria in a flow-through environment. The entire system is controlled by a microcomputer that interfaces with the valves and other controlling equipment via microprocessor-based data acquisition and control hardware. This allows FATES to be run at varying temperatures, salinities, suspended sediment loads, and water flow-through rates. Currently, the system may be simultaneously run at two different temperatures (in blocks of 12 aquaria each), fresh and/or one salinity (in blocks of 6 aquaria), 24 different suspended sediment levels, and 24 different water flow-through rates. The system also incorporates a timed light-level control for day/night simulation.

138. Water flow-through in FATES is controlled by electric solenoid valves that allow 600 ml of water to flow into each aquarium every 2 min. This equals 95-percent replacement of the aquarium water every 12 hr. The system also contains manual valves that allow water to flow into the aquaria constantly for flushing between experiments. The volume of flow-through is controlled by the computer program and can be set at any volume needed within the constraints of the water supply system.

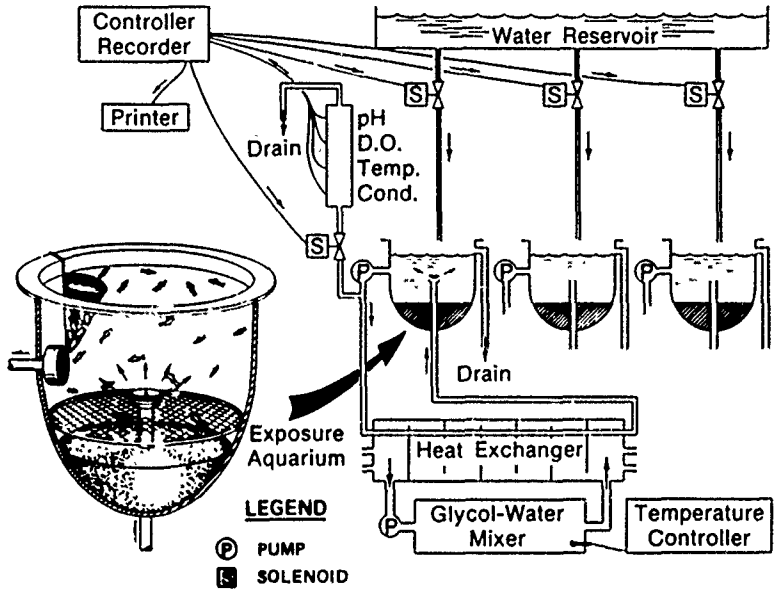


Figure 5. Schematic diagram of the Flow-Through Aquatic Toxicology Exposure System

139. All three apparatus will provide the desired quality of performance and, at present, are the devices for flow-rate control that can be recommended for dredged material bioassays.

Aeration

140. Where supplemental aeration must be provided, simple mechanical aquarium air pumps are usually satisfactory. Compressor units that use oil-lubricated cylinders must not be used unless they are followed by a membrane filter, and this in turn followed by bubbling the filtered air through a water column. Other equipment that can be proven to be noncontaminating may also be acceptable.

Water Supply

141. There are three types of sources from which water suitable for conducting dredged material bioassays may be obtained. The source of preference, as recommended in the Implementation Manual (USEPA/USACE 1977), is water drawn

from the disposal site area. If this is not practical, as is often the case, uncontaminated natural or artificial water prepared according to accepted chemical formula is acceptable.

142. An example calculation of the approximate volume of water required for the conduct of one solid phase bioassay using a polychaete worm, a clam, and a shrimp species is shown in Appendix F. It is based on the assumptions that both a reference sediment and a control sediment are used in addition to the dredged material, and that all species will be tested simultaneously. In the example, five replicates of each treatment are established in 10-gal aquaria with a once-through flow of water equal to 6 aquarium volumes per 24 hr. Based on these assumptions, the volume of water that will be required is large, ranging from almost 22,000 to more than 43,000 gal. The lower figure is calculated base on three species being tested, with the two compatible species (the polychaetes and the clams) being tested together. The higher figure would result if a fourth organism were included and all species were tested separately. In addition to this is the volume required for the holding facilities, which may or may not be large, depending on whether the facilities are static or flow-through.

143. The Corps project manager should bear these volumes in mind when judging whether or not a contractor actually has the ability to meet the required specifications. Very little water is actually required to conduct static liquid phase and suspended particulate phase bioassays. These tests are most likely within the capabilities of any contractor who can satisfy the other requirements involved. The large quantities of water required to conduct flow-through solid phase bioassays can be prohibitive to a contractor who must rely entirely on artificial water or who must truck and store large volumes of natural water.

144. A synthetic seawater suitable for culturing and testing marine and estuarine organisms can be made from balanced mixtures of salts and trace elements available commercially. These salt mixtures closely parallel the natural salt balance of the ocean but also contain a small amount of EDTA (ethylenediaminetetraacetic acid) to prevent precipitation of certain cations, which are otherwise difficult to solubilize but are necessary to achieve the correct ionic balance. High-salinity water may be used to increase the salt content of fresher water if necessary. However, test water should not be made

entirely from natural sea salts that have been dried, as they may not regain their original capacity to support life.

Water Quality Testing

145. Temperature, pH, dissolved oxygen (DO), ammonia, and salinity, if appropriate, should be monitored in the test aquaria and recorded at least once daily. Ammonia in bioassays is an important water quality parameter. Therefore, ammonia should be carefully and judiciously monitored. In bioassays, there are two sources of ammonia: waste product from the test organism and microorganisms in the test sediment. The latter can be considerable in highly organic sediments. Jones and Lee (1988) reviewed a large volume of bioassay data and concluded that ammonia, not contaminants, may be largely responsible for much of the toxicity observed. Water quality data sheets should be standardized and should include date, name of sampler, time of day, aquarium identifier, and comments and observations.

146. Portable, low-cost instrumentation is available, making it possible to take direct measurements in the test aquaria. A combination portable electronic thermometer and DO sensor is a common instrument, and every bioassay laboratory should have at least one. Documentation of the calibration of all instruments used should be kept in a single logbook and should be available for inspection by the Corps project manager on request. (See Jones and Cullinane (1985) for calibration recommendations for some major laboratory equipment.) Oxygen-sensor membranes should be changed on the schedule recommended by the manufacturer, and recalibration should follow each change after conditioning the membrane for a time by soaking in water. All of these operations should be recorded in detail in the instrument calibration logbook.

147. Remote sensing telethermometers are available which are capable of continuously monitoring up to 12 thermistors. These probes may be purchased in almost any length. One such instrument with the probes randomly distributed among the aquaria in a sediment bioassay array would provide an excellent verification of the diurnal temperatures in the aquaria. Such an instrument is to be recommended, especially in the case of the "table-top" bioassayist who relies solely on control of ambient air temperature for the maintenance of aquarium water temperature control.

148. Portable, hand-held digital and analog pH meters are becoming smaller and less expensive with no sacrifice in accuracy. These should be standard instruments in the bioassay laboratory. As with other instruments in frequent and routine use, they must be calibrated at regular intervals, and all calibration data kept in the instrumentation logbook. Calibration should be done using standard buffer solutions. The buffers should be discarded after one usage. At least biweekly, the meter readings should be checked against a standard buffer solution. Batteries in portable models should be replaced at 2- to 3-month intervals, or more often if leakage is detected.

149. Two types of instruments are available for measuring salinity. One is the portable conductivity-salinity meter that also contains a temperature-sensing circuit for converting the conductivity reading to a salinity reading; the second type is the temperature-corrected optical refractometer-salinometer. Both are equally accurate and within the same price range. The choice is up to the contractor. These are the basic instruments that the bioassay contractor must possess and the basic water quality parameters that must be routinely measured.

150. Supplemental aeration should not ordinarily be required during dredged material bioassays. The most likely exception to this is in the case of static tests conducted with fish. The flow-through bioassays should continually bring in sufficient aerated water to keep the DO concentration above 5 ppm. If the DO drops below 4.0 ppm, supplemental aeration should be initiated at once.

151. Optimally, pH in seawater systems should remain stable near the range of 7.5 to 8.5. Although marine organisms can tolerate gradual decreases in pH to 7.0 or below without direct harm, decreasing pH can signal possible harmful deterioration in the water quality. Oxidation reactions are all acid-forming, which results in an increase of carbon dioxide in the water, thus decreasing the pH. The presence of excessive uneaten food or dead organisms in the water could cause a bloom of heterotrophic bacteria and the consequent release of large amounts of carbon dioxide into the water. Increasing pH is often observed when algal populations are large. For reasons such as these, stable pH readings are important indicators of good water quality and support given to the assurance of a properly conducted test.

Sediment Homogenization

152. The volumes of sediments required for the performance of a single complete three-phase sediment bioassay are not large (see Appendix G). It is therefore essential to the validity of the tests and the verity of the replications that the composited sediment samples are completely homogenized. There are numerous methods for achieving homogenization, and the Corps project manager must be familiar with the methods of an individual contractor and observe the process in action in order to make a judgment regarding its efficacy. As much latitude as possible should be given to the potential contractor in devising means for achieving sediment homogeneity. However, the importance of thorough sediment homogenization to the success of a high-quality sediment bioassay cannot be overemphasized. Sediments that contain toxic materials such as PCBs should be treated with caution to reduce exposure to the laboratory workers. (See Appendix H for guidelines for handling contaminated sediments.)

153. The first step in the process is to decant any supernatant water from the collection containers without disturbing the settled sediment. The sediments to be homogenized must then be passed through a 1.0-mm screen to remove debris and any resident macroorganisms. A squeegee (a T-shaped rubber-bladed tool) can be used to break up lumps and aid in moving the sediment through the screen. The sediment is then mixed with test water in a volume exactly equal to 4 times the volume of sediment, thus giving a sediment-to-water ratio of one to four (Vol/Vol).

154. When the container is approximately two-thirds full, the contents are slurried completely, either by agitation on a large shaker platform or by mixing rapidly using a high-powered industrial-type portable mixer with a 316 stainless steel shaft and propellers or preferably a 316 stainless steel high-shear impeller. The slurrying process is performed for 30 min, after which the suspension is allowed to settle for 1 hr (residual solids used in solid phase bioassay); then, the supernatant is decanted, pumped, or siphoned off. If the sediment thus prepared is the dredged material under investigation, the supernatant is stored in containers prepared as instructed in the Implementation Manual (USEPA/USACE 1977). If the sediment is the control or reference material, the supernatant is discarded.

Liquid and Suspended Particulate Phase Tests

155. The sediment required for conducting liquid phase and suspended particulate phase tests is relatively small and can be estimated by using the procedure outlined in Appendix G of this document. Sufficient supernatant should be retained after the 1-hr settling period to ensure adequate material to conduct all of the liquid and suspended phase tests. Allow for some loss or wastage, and enough material for chemical analysis of the liquid phase material if required. A vessel capable of holding all of this material must be available and equipped with a stirrer capable of rapidly recreating a homogeneous suspension of the fine-grained material that will not have settled out of the original suspension after 1 hr, or the vessel must be capable of fitting on a large shaker table that would achieve the same purpose of resuspending the material. Stirring or shaking is continued only until the fine-grained material is resuspended. This should not take more than a few minutes. When resuspension is complete, manually handleable aliquots are drawn at once from a valve in the low side or the bottom of the mixing tank into suitable containers such as 1-gal glass jars.

156. At this point, about one half of the containers may be stored in the cold room until required for the suspended particulate test, if one is to be conducted. No further treatment is needed except to thoroughly resuspend the settled sediment particles and make the necessary dilutions just prior to initiation of the tests. The particulates must be removed from the remaining sediment if an elutriate is to be obtained for the liquid phase test. This can be accomplished by pressure filtration through a series of progressively finer cartridges, or more commonly by initial centrifugation followed by filtration. For these procedures, some special equipment is required.

157. If centrifugation is to be used, the contractor will require a centrifuge capable of containing four to six 0.5- to 1.0-ℓ centrifuge bottles, and operating at speeds of 3,000 to 5,000 rpm. Centrifuge bottles composed of 316 stainless steel are preferred. Disposable plastic centrifuge bottles should not be substituted. These plastic bottles are usually composed of polypropylene, and the disadvantage in using them is that hydrophobic organic pollutants and some metals which may be in solution in small quantities tend to adsorb to the surfaces of such materials and may be lost from solution, thus reducing any toxicity of the liquid phase.

158. After centrifugation, the decanted supernatant must be filtered. This is accomplished in two steps: prefiltration and final filtration. The prefiltration step is intended to remove the bulk of any remaining suspended material that would clog the very fine 0.45-μ filter used in the final filtration. If the sediment is not predominantly fine-grained, the prefiltration step may be unnecessary.

159. Two methods are available for accomplishing these filtrations: suction filtration and pressure filtration. Suction filtration involves using a suction pump to create a vacuum within a suction flask to which a filter funnel is attached, or within a manifold containing a series of filter funnels. The equipment is inexpensive but, considering the volumes involved, it is highly labor-intensive and is not the preferred method.

160. The second method, pressure filtration, involves forcing the centrifuged supernatant through a prefilter cartridge and finally through a 0.45-μ membrane-filter cartridge. High-purity nitrogen gas is used to pressurize the system. Size 1a gas bottles and a two-stage regulator are required, and the pressure chamber is a 316 stainless steel unit of at least 5-gal capacity. Filter holders, filter cartridges, and some interconnecting plumbing comprise the remainder of the system. Filtration can be accomplished rapidly using this type of equipment, but it is more expensive than suction filtration equipment.

Additional Laboratory Instrumentation and Support Equipment

161. Every bioassay laboratory must be equipped with such an array of miscellaneous tools, glassware, and small instruments that it would be pointless to attempt to enumerate them and would only add to the confusion of an individual functioning in the capacity of a Corps project manager. This being the case, only a few of the major essentials, other than those so far described, will be discussed here. Suffice it to say that sufficient routine equipment and supplies, repair and replacement parts, reagents, etc., should be on hand to minimize delays in the testing when the inevitable unexpected mishap occurs.

162. The cleaning of glassware and glass aquaria is critical to the conduct of reliable bioassays, and considerable care and thought should have been given to the preparation of the cleanup area. Several types of solvents

will be used in the cleaning processes, some of which are volatile or corrosive. Careful attention must be given to technician safety. The most desirable physical fixtures are large-capacity stainless steel double sinks with generous drain boards and a ventilated hood covering the sinks. The hood is necessary because volatile solvents such as acetone and hexane will be used at times as well as dilute hydrochloric acid and hypochlorous acid. The other solvents will be hot and cold tap water and deionized or glass distilled water. Detergents may also be used. The Corps project manager should be concerned with the cleanliness and general condition of the cleanup area. It should be well ventilated, not dusty, and well removed from the area in which sediments are processed. The glass distillation apparatus should have a capacity of 4 gph or more and should operate into an automatic still tank. Distilled water should be available from a tap at the sink on demand.

163. Weighing balances (top-loading, pan or electronic) are essential for conduct of bioassays. Weighing balances will be used for everything from taking the weights of experimental organisms to balancing centrifuge bottles for preparation of the liquid phase medium. Electronic balances are recommended for bioassay testing because they are more accurate and easily manipulated. Calibration of all balances should be done annually by a certified balance technician, and these records should be made available. In addition, a monthly check should be made by laboratory personnel using weights traceable to NIST standards. These should be recorded in a permanent logbook. Lightboxes, consisting of low-wattage fluorescent tubes under a frosted glass panel and illuminated magnifiers, are necessary tools for counting and observing very small organisms. A low power (6 to 50 ×) stereo-binocular wide-field microscope is also useful in this regard as well as for identification of organisms. A compound microscope and hemacytometer are needed for algal work.

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PART V: MANAGEMENT OF CHEMICAL ANALYSIS CONTRACTS

164. This section of the document provides the project manager with guidance on the wide variety of activities required for management of chemical analysis contracts. Detailed guidance for field personnel is provided for field spiking procedures since this area is not covered in detail in any of the available reference manuals.

Minimum Field and Laboratory Capabilities Required

Facilities and equipment

165. A contractor's facilities should be of adequate size, with satisfactory lighting, ventilation, constant temperature, low noise levels, and low humidity. Services such as electricity, water, and air should be present and operational. General field equipment must be present in sufficient quantities. This includes equipment such as thermometers, water and sediment sampling apparatus, pH meters, dissolved oxygen (DO) meters, and flowmeters. General laboratory equipment must be present in sufficient quantity and condition to provide environmental analyses having the quality and integrity established by the Corps project manager. This includes such items as air conditioners, drying ovens, ventilation system, furnaces, generators, refrigerators, incubators, laboratory hoods, sinks, counters, and analytical instrumentation as required by the analytical methods used. Cleanliness in the laboratory should be emphasized to reduce possible contamination. Also, incompatible analyses should not be performed in the same work area.

166. Safety features to comply with Federal Occupational Safety and Health Administration regulations should be readily available. These include fire extinguishers, safety showers, eyewash stations, mandatory eye protection requirements, first aid equipment, protective garments, chemical dispensing devices, and safety education.

167. Sediment samples collected in the field may contain toxic materials, such as polychlorinated biphenyls (PCBs), and thus should be treated with caution to reduce occupational exposure to workers. General guidelines for handling contaminated sediment are presented in Appendix H. These guidelines do not purport to address all safety problems, but they can serve as a guide for writing appropriate safety procedures. It is recommended that the

contractor develop appropriate safety procedures that will meet the need of a specific project. For additional information on toxicity to humans and recommended handling procedures, see Green and Turk (1978).

Personnel

168. Personnel qualifications and experience should be commensurate with the difficulty and complexity of the chemical analyses to be performed. Some analyses require no sample treatment, and the measurement can be performed in minutes on a simple instrument. Other determinations require extensive sample preparation prior to complex instrumental examination. Presented in Table 9.1 of the "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (USEPA 1988) are skill ratings for a range of standard analytical operations. Such skill ratings are useful when assessing the adequacy of contractor personnel to reliably complete required chemical analyses of similar complexity. The analyses usually required by the CE in their dredged material testing are complex and require experienced analysts comparable to GS-9 through GS-12 with backgrounds in analytical techniques.

169. Successful implementation of a quality assurance plan depends on the competence of the employee during the chemical analysis. All employees should have training in their appointed jobs to contribute toward producing a high-quality product. Several methods of training are discussed in the "Handbook for Sampling and Sample Preservation of Water and Wastewater" (USEPA 1982). Laboratory supervisors should periodically review techniques and policies of the contract with the analysts, stressing the importance of applying quality control procedures on a continuous basis.

Management of Field Operations

Quality assurance coordination

170. The use of Government quality assurance samples must be the discretionary decision of the project manager, but generally should be limited to approximately 10 percent of the total number of samples for laboratory analysis. Prior to field sampling, the project manager, the field crew chief, and the laboratory quality assurance officer should meet to coordinate the implacement of field-prepared reagent blanks, standard reference materials, split samples, and spiked samples in each sample lot, and to divide daily samples into subparts in which quality control samples can be randomly placed.

Placement of the samples should be designed to divide the daily sample run into sublots so that, in the event that errors in the laboratory or field are discovered in analytical data, the entire sampling run would not have to be repeated. Spike only samples for which an unspiked duplicate is also included. (Unless absolutely assured of the representativeness and homogeneity of a sample divided into three aliquots, a spiked sample should not be regarded as more than a check on chemical degradation of a sample.) The frequency with which samples should be split or spiked and a schedule for submission of reference material are discussed in the section on Government quality assurance (paragraphs 195-206).

Sample containers

171. Basic to field quality assurance is the use of proper sample containers appropriately cleaned. When choosing proper sample containers, things to consider include parameters to be analyzed, durability of containers, container resistance to breakage, size, weight, adsorption and/or desorption tendencies, and cost.

172. The two major sample container materials are glass and plastic. Borosilicate glass is recommended where glass containers are needed, and linear polyethylene is recommended where plastic containers are used. Teflon containers would be selected for use above polyethylene were it not for the prohibitively high cost.

173. Teflon or polyethylene container caps can be used except where samples are taken in glass for organic analysis (e.g., pesticides, oil and grease). Container caps for glass bottles should be made of or lined with Telfon. Aluminum foil cap liners have been used in the past, but these are not recommended for use because of their propensity to tear.

174. Wide-mouthed sample containers are recommended because they permit easy sample access and cleaning. Narrow-neck bottles are recommended when there is a need to minimize contact with cap liners or the outside atmosphere.

Cleaning procedure
for sample containers

175. Every laboratory has specific procedures for washing and preparing sample containers. The following procedures for washing and preparation of containers are offered as general guidelines for use (USEPA 1982):

a. Wash containers and caps with nonphosphate detergent (e.g., Liqui-Nox) and scrub strongly with a brush. (If possible, wash liners and caps separately.)

b. Rinse with tap water, then distilled water.

c. Invert to drain and dry.

d. Visually inspect for any contamination prior to storage.

e. If container requires additional cleaning, rinse with a chromic acid solution (35 ml saturated sodium dichromate solution in 1 ℓ of sulfuric acid) (this solution can be reused). Then rinse with tap water and distilled water and dry as indicated above. A commercial product, NOCHROMIX, may be used in place of chromic acid.

176. For certain parameters, a special cleaning procedure is needed to avoid adsorption or contamination due to interaction with container walls. These procedures are:

a. Metals and phosphorus. If metals are to be analyzed, rinse the container with a solution of one part nitric acid to four parts water, then with distilled water. If phosphorus is to be analyzed, rinse the container with a solution of one part hydrochloric acid to one part water, followed by distilled water.

b. Organics. If oil and grease or pesticides are to be analyzed, rinse the sample glass container with methylene chloride, followed by acetone. The container should have been previously cleaned with chromic acid solution, as described previously. Treat the container cap similarly.

c. Precleaned bottles. As an alternative to washing and cleaning bottles in the laboratory, precleaned bottles that meet USEPA specifications may be purchased from commercial vendors.

Preservation techniques

177. Preservation methods are limited in their effectiveness and are intended to:

a. Retard biological activity.

b. Retard hydrolysis of chemical compounds and complexes.

c. Reduce volatility of constituents.

Most of the preservation methods used today are a form of chemical addition, pH control, refrigeration, or freezing. Appendix D contains guidance concerning preservation methods for particular analyses as outlined by Battelle (1988). At its best, sample preservation is very difficult because almost all preservatives exhibit some interferences on particular analyses. Storage at low temperature (4° C) is perhaps the best way to preserve most samples for

short periods (<24 hr). Alternate preservation methods in Appendix D could possibly be required in addition to storage at low temperature.

Field reagents

178. Reagent and solvent purity used in sampling operations must be equal in quality to that used in the analytical laboratory. All acids used for field spiking and preservation of water samples for metals analyses by atomic absorption must be analytical reagent grade or better. Even analytical reagent grade acid may require low-temperature distillation in borosilicate glass to remove background metals content to below instrumental detection limits. Acids should be distilled before use if necessary.

179. The minimum purity of reagents that can be used for organic analysis is analytical reagent grade (USEPA 1981). Due to the great sensitivity (nanogram and subnanogram quantities) of gas chromatography (GC) used to quantitate organics, maximum purity is frequently required. The specificity of some GC detectors requires that reagents and solvents be free of classes of compounds to be analyzed. For example, analyses by electron capture require that reagents and solvents be free of electronegative materials that would interfere with the determination of specific compounds in the sample. Pesticide-quality solvents, available from several sources, are required when doing low-level work such as for tissues and water. Analytical reagent grade solvents are permissible when analyzing sediment samples.

180. Standard Reference Materials, presented in Appendix I, and certified check samples are available from the National Institute of Standards and Technology (NIST) and the US Environmental Protection Agency, respectively. These samples should be prepared by the laboratory quality assurance officer and given to the contractor field crew chief for random placement among field samples. Objective treatment of such samples is admittedly difficult to obtain when contracting both field sampling and laboratory analyses from the same contractor. Therefore, results obtained from check samples used in this fashion should be limited in use to checks on sample degradation and contamination resulting from sampling and preservation.

Distilled water quality

181. The importance of purified water (distilled, deionized) is realized in its many uses, e.g., preparation of reagents and standards, dilution of solution, and final rinsing of glassware. In addition to reagent water blanks being analyzed with all sets of analysis, the laboratory water must be

checked periodically (2 to 4 weeks). The methods used to check the water may consist of electrical conductivity and resistivity, potassium permanganate ($KMnO_4$) color retention test, total organic carbon, and others depending upon the analyses being performed. These procedures are described in the USEPA (1983) and ASTM Methods (1988). Reagent water prepared in the laboratory for field dilutions should conform to ASTM Specification Type I or Type II (USEPA 1988).

Field spiking

182. Field preparation of reagent blanks, spiked samples, and split samples is contamination prone; however, there are few substitutes for determining sample degradation occurring after removal from the field environment and prior to analysis, or contamination resulting from preservatives, distilled water, containers, or sample handling. The purpose of spiked samples is primarily to determine the degree to which the sampling, sample handling, shipment, and storage affect sample representativeness of the field site. Split samples provide a check on water column sample homogeneity, the basis for determining spike recovery, and, possibly, laboratory analysis precision.

183. It is essential to the validity of the field sampling technique and the verity of field spiking that the sediment/soil samples are completely homogenized. Therefore, due to the difficulty and improbability of preparing a thoroughly homogeneous sample, field spiking of sediment/soil is not recommended. Conversely, water samples are relatively homogeneous with respect to dissolved constituents, while particulate matter may be unevenly distributed. Notwithstanding the different effects that the aqueous and particulate phases may have on the spike, the purpose of the procedure is to determine the overall effect of sampling procedures and storage on the sample with respect to the chemical parameters to be analyzed. Phase distribution can be determined in the laboratory if substantial differences are found between split samples or if theoretical versus actual spike recovery percentages occur.

184. Spiking solutions are purchased or prepared in the laboratory from primary standards dissolved in either distilled water or a water-miscible organic solvent for field use. Successful use of this procedure presupposes that spiking is performed by the field crew chief using acceptable volumetric transfer techniques and that identification of the spiked sample, the spike concentration, and associated duplicate and reagent blanks are permanently recorded in the field logbook.

185. Spikes are added to measured volumes of samples appropriately preserved for the parameters to be analyzed. Organic samples are devoid of preservatives, while metal samples must be preserved with either ultrex (or equivalent) hydrochloric or nitric acid to a pH of 2.0. A clean volumetric pipette must be used to quantitatively transfer the spike from the stock reagent to the premeasured sample. A description of the addition of a cadmium spike is provided in Appendix J for illustrative purposes.

Reagent blanks

186. Prepared reagent blanks from the field should be submitted with all split and spiked sets of samples. The purpose of the blank sample is to provide a check on field degradation of reagents as a function of time, exposure to contaminants from handling, sample container, etc. Each blank is composed of distilled water and the appropriate preservative for each type of sample to be analyzed (e.g., for metals, distilled water and few drops of HNO_3 or HCl to give a pH of 2.0). Adequate sample sizes should be provided for the laboratory procedure to be performed on each sample type. Sample numbering, storage, field log notation, and further handling must be identical to that accorded other samples (USEPA 1986).

Tissue spiking and duplication

187. These procedures are performed in the laboratory using methods prescribed by the appropriate analytical procedure.

Analytical Methods

188. Acceptable analytical detection limits for water and tissue samples should be specified by the CE in the contract. All procedures, unless authorized in writing, are to conform to the guidelines established in the publication "Ecological Evaluation of Proposed Discharge of Dredged Material Into Ocean Waters" (USEPA/USACE 1977). It is mandatory, however, that the latest editions of the referenced method manual be used when conducting the procedures. For example, the USEPA manual "Methods for Chemical Analysis of Water and Wastes" of 1979 has been superseded (USEPA 1983).

189. The exact procedures to be used should be specified by reference in the contract since there may be several approved methods for the same parameter. Copies of these procedures should be readily available in the laboratory for each analyst's use. Analytical precision and accuracy will vary

depending on the kind of sample (sediment, tissue, water) and the chemical parameter being analyzed. Contractors who have previously conducted the required testing should be able to provide precision and accuracy data for their laboratory. If they have not previously conducted such tests, preaward testing may be necessary.

Management of Laboratory Operations

Contractor's quality control program

190. Internal quality control. The contractor should have a written internal quality control program that is available to the project manager for review and retention. The quality control program should be comprehensive, and should include discussions of calibrations, instruments, equipment, reagents, supplies, and analyses. The following quality control practices are the minimum requirements for the contractor's internal program:

a. Fifteen to twenty percent of the analytical effort is to be devoted to internal quality control analyses. Any work effort involving less than five analyses will necessitate one analysis as a quality control analysis.

b. Quality control analyses include replicate (analysis of one sample twice), percent recovery, and reference material. Percent recovery analyses may be undertaken in the following ways:

(1) Conducting analyses of a sample that has been spiked with a known amount of measured material. A reagent blank is not to be considered a sample when spiking. The percent recovery is to be calculated according to the following equation:

$$\text{Percent recovery} = \frac{(\text{Sample} + \text{Spike}) - \text{Sample}}{\text{Spike}} \times 100$$

(2) Analyzing a known concentration of a standard reference material or quality control check sample that has been processed exactly as the sample(s). The percent recovery is then calculated according to the following equation:

$$\text{Percent recovery} = \frac{\text{Analyzed value}}{\text{Referenced value}} \times 100$$

Analyses of environmental samples usually entail significant background interference problems. Therefore, the analysis described in paragraph 190b(1) is the preferred percent recovery method. Evaluation procedures for replicate and percent recovery analyses are presented in Chapter 6 of the "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (USEPA 1988).

c. Reagent blanks prepared in the laboratory are to be analyzed on every analytical run. A reagent blank is not to be considered as part of the percentage devoted to quality control analyses. The method blank is defined as an appropriate volume of distilled water, free of contamination from the parameter being determined, which has been processed exactly as the sample (including glassware, reagents, solvents, etc.).

d. A new standard curve should be established with each new batch of reagents, using at least seven concentrations for atomic absorption analyses. Thereafter, at least a three-point calibration curve is to be developed each time a discrete group of samples are analyzed using atomic absorption analyses. Analyses of PCBs, DDT, and specific petroleum hydrocarbon compounds using gas chromatography are not conducted using standard curves. For these analyses, linearity of the detectors on the gas chromatograph should be checked monthly.

e. Provisions should be included in the contract for use of the standard addition technique (Friedman and Erdmann 1980) for atomic absorption analyses when interferences cannot be avoided or are unknown. Standard additions are used for all flameless and heated-vaporization atomic absorption methods.

f. A standard or reagent blank should be inserted (in random order) at every seventh or eighth sample or as specified in the method. If there is a difference of over 2 percent for metals or 10 percent for organics from the initial standard readings or if there is noticeable baseline drift, the instrument should be recalibrated, and all samples run after the last acceptable calibration check should be reanalyzed.

g. The contractor should submit reference material (NIST or USEPA) into his own laboratory system on a regular basis. A minimum of two reference materials for trace inorganic constituents and one reference material for organic compounds should be submitted weekly (Friedman and Erdmann 1980). Quality control charts should be constructed using the results of the reference material analyses. Quality control charts include an expected value and statistically determined "warning" and "control" limits (Friedman and Erdmann 1980). Control charts indicate trends and variability in analyses which may not be readily apparent from an examination of tabulated data. They can effectively determine if bias is developing or if precision is less than expected. Warning limits are generally established at 2 times, and control limits at 3 times the standard deviation of the statistic used. Either individual or average values can be used in control charts.

Detailed procedures for setting up and interpreting quality control charts are given by Friedman and Erdmann (1980). In general, analyses must be stopped to identify and resolve the problem when any result is beyond the control limits or when seven successive points are on the same side of the expected value (USEPA 1988). When a problem is identified, several samples throughout the analytical run must be reanalyzed, including samples near the beginning, the end, and on either side of the reference sample. When the extent of the problem has been identified and its solution documented, analyses throughout the problem area are repeated, and the data are corrected.

191. Special care must be taken when analyzing trace organics such as PCBs, DDT, and petroleum hydrocarbons. The following quality control measures should be followed by the contractor during gas chromatographic analyses of these parameters. A more detailed discussion is provided in the "Manual of Analytical Quality Control for Pesticides" (USEPA 1981).

a. All samples and/or standards are to be injected by the "Solvent-Flush Technique" when an autosampler is not available.

b. Special note should be made of the assay of the less pure materials available for use in standard solutions. Every effort should be made to obtain the best grade available.

c. Precautions are to be taken to maintain the integrity of the reference materials. These would include freezing and refrigeration, use of proper storage containers and conditions to maintain concentrations, and protection from incident ultraviolet radiation.

d. Stock and standard solutions should be monitored for deterioration by dating material, checking for physical alteration, and analytical checking against fresh standards solutions.

e. Solvents are to be redistilled in glass to remove any interfering substances. Distilled water used for control samples should be preextracted and then boiled to remove volatile organic interferences and solvents, respectively.

f. Injection port, column oven, and detector temperatures must be accurate and constant at levels specified in the specific method.

g. The linearity range of the detector must be determined by the laboratory and operation confined to this range. The manufacturer's suggested linearity range may not be representative of the linearity range actually obtained in the laboratory.

h. After the optimum operating conditions are defined, they must be sustained through a routine maintenance program. This includes septum changes, temperature monitoring, tank and filter replacement, and other common laboratory practices.

i. System performance should be monitored on a daily basis by running standards and comparing elution patterns, relative peak proportions, peak geometry, peak intensity, and relative retention times. If these parameters change from day to day, then corrective action should be taken. If uncorrectable, they should be construed as indications of contamination from a deteriorating column or detector that must be replaced.

192. Calibration and maintenance program. Regardless of the instrument and equipment types, a system of calibration and preventive maintenance is necessary. The QA/QC plan should specify maintenance requirements and schedules, and the regular maintenance should be documented upon completion.

193. Potential contractor laboratories should be checked to determine, where possible, if laboratory equipment is routinely calibrated using standards traceable to NIST standards. For example, analytical balances should be calibrated using weights certified by the NIST. Where NIST calibrations or standards do not exist, the contractor should maintain a contract with the manufacturer or have regularly scheduled service to maintain instruments at manufacturer-specified levels. Each instrument should also be assigned a unique identification number. Documentation shall identify the specific instrument, where and when used, maintenance performed, and the equipment and standards used for calibration.

194. All laboratory instruments need calibration and periodic servicing. Calibration recommendations for some major laboratory instruments can be found in Jones and Cullinane (1985). These recommendations should be viewed as guides with the intention of giving the laboratory manager an idea of what type of service is needed and approximately how often.

Government quality assurance

195. General. In addition to verifying that a contractor's internal quality control procedures are adequate, the project manager is responsible for ensuring that testing activities are implemented to monitor its performance. This effort should constitute approximately 10 percent of the total analytical program.

196. Contractors, however, are normally responsible for all aspects of a dredging study. This results in the contractor becoming responsible for spiking and splitting of field samples. Sample spiking and splitting should be required of the contractor at the same rate as if the Government were sampling and then submitting samples for chemical testing. If a contractor is conducting all phases of a study, the spiking and splitting of field samples

essentially becomes part of the contractor's quality control program. This type of testing should be in addition to the 15 to 20 percent effort devoted to the contractor's internal quality control effort. The Government's quality assurance testing will therefore consist almost exclusively of reference material analyses.

197. Every contract should specify that at the beginning of each project, or when dealing with a new contractor, the comprehensive quality assurance program presented in Chapter 6.5 of "Handbook for Analytical Quality Control" (USEPA 1988) be carried out for each parameter in the study. This program of replication, split samples, and spikes involves only seven samples for each parameter, but should be invaluable for identifying problems in the field and laboratory prior to full-scale sampling and analysis.

198. If a laboratory is certified for analysis of drinking water by the USEPA or a state, it will already be engaged in quality assurance round robins and will be receiving check samples for analysis on a regular basis. This in no way lessens the responsibility of the project manager to assess the continuing capability of the contractor with tissues, sediments, and disposal site water in which analytical interference problems may be different and more severe than with drinking waters.

199. The interlaboratory testing activities presented in the following sections are adaptations for Corps use of procedures used by the US Geological Survey (Friedman and Erdmann 1980) and the USEPA (1988). These procedures include analysis of reference materials, split samples, and spiked samples. The project officer should ensure that the following quality assurance procedures are carried out for every project under his supervision.

200. Reference material analyses. Standard Reference Materials (SRMs) for trace metals and inorganic and organic constituents can be obtained from the NIST. These are well-characterized materials produced in quantity to improve measurement techniques. The SRMs are certified for specific chemical or physical properties, and issued with certificates that report the results of the characterization and indicate intended use. These materials help develop accurate methods of analysis, help calibrate measurement systems, and help establish quality control. Appendix I lists SRMs for trace metals and other inorganic constituents in water (SRM 1643b), estuarine sediment (SRM 1646), Buffalo River sediment (SRM 2704), and bovine liver (SRM 1577a).

Standard Reference Materials for organic constituents are also presented in Appendix I.

201. Ampulated concentrates of trace metals and other inorganic constituents as well as PCBs in fish and sediment are also available from the USEPA, Quality Assurance Branch, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268. The latest edition of the USEPA Quality Assurance Newsletter, which is published semiannually, should be obtained for details on the QC check samples. To ensure that proper quality assurance guidelines are followed, the CE personnel should be responsible for quantitatively preparing solutions from the concentrates using a deionized-water matrix and, if feasible, a natural-water matrix. In the latter case, both the sample spiked with the concentrate and the unspiked sample must be analyzed by the laboratory.

202. One reference sample should be submitted for every 25 samples analyzed. If in any month 10 or more samples are analyzed, submit a reference sample. Reference materials should be placed among the samples submitted. If possible, submit reference materials in such a way that the receiving laboratory will not know they are reference materials. If this is not possible, i.e., when the contractor collects and analyzes the sample, ensure that the contractor is not informed of concentration values prior to sample analysis. Reference materials are generally provided with an expected value and standard deviation. These values can be used by the project officer to determine if the contractor laboratory is performing with acceptable accuracy.

203. Spiked sample analysis. Every twenty-fifth sample collected (or one sample per month if between 10 and 25 samples are analyzed in a month) should be spiked with a known concentration of the constituent(s) to be analyzed. Submit both spiked and unspiked portions to the contractor. Final concentrations of spiked samples should be in the same range as concentrations expected in the samples.

204. If spiking with more than one constituent may cause interference problems (such as coprecipitation) or if the contractor collects his own samples, provide the material to be used for the spikes directly to the analyzing laboratory. In no case should the laboratory or cooperating agency be informed of the concentration of the resulting spike prior to the analysis. Appendix J contains an example of a procedure for spiking water with cadmium.

205. Split sample analysis. Split samples provide a way of checking the precision of a contractor and may also be used to check the comparability of results between contractors. A split sample is a collected sample that is thoroughly homogenized (especially important for sediment and tissue samples) and evenly divided into two or more subsamples. Each subsample is then analyzed as an independent sample. The samples may be analyzed separately by the same laboratory or analyzed by two different laboratories as a check of the analytical procedure or comparability of results.

206. Every thirtieth sample should be split into a minimum of two subsamples. Additional split samples may be generated if it is desired to send split samples (minimum of two) to additional contractor or referee laboratories. In any month in which 10 or more samples are analyzed, submit split samples even though less than 30 samples are submitted. If possible, submit samples in such a way that the contractor laboratory will not know that they are splits of the same original sample, e.g., by disguising the name of one sample. If the contractor collects his own samples, submit the split samples to the contractor in addition to the regular samples analyzed. Split samples should be used with tissue, sediment, and water samples.

Evaluation of Government quality assurance testing

207. If the contractor is responsible for all phases of the dredged material testing, the contractor will know the identity of reference samples and put forth his best efforts on such samples. The project manager therefore has cause for concern if the value reported by the contractor for a reference material differs substantially from the expected value. To determine when substantial differences exist, the project manager should maintain quality control charts for the reference materials submitted to a contractor. See Friedman and Erdmann (1980) for detailed instructions on quality control charts.

208. When problems with the analyses are not amenable to rapid solution, it is good policy to use a referee laboratory. To use a referee laboratory, the project manager should submit aliquots of identical split and spiked samples to the contractor and to the referee laboratory for analysis. If both the contractor and referee laboratories report similar results, it is probable that the field crew is in error (i.e., improper spiking procedures, weighing errors). If the referee laboratory, however, produces data that are

acceptable but the contractor laboratory does not, then the problem lies with the contractor and should be quickly resolved. If the splitting procedure itself causes analytical differences, a fact that should be proved or disproved early in a study by analysis of split samples, use of unknown spiked samples or laboratory inspection will be required to resolve conflicts. The use of referee laboratories when analytical problems arise should be a requirement placed on the contractor in the scope of work.

Cost-reduction techniques

209. The ecological fate of contaminants contained in dredged material is of great concern to the CE and regulatory agencies. Disposal of contaminated dredged material may adversely affect water quality and aquatic, wetland, or terrestrial organisms. Therefore, it is very important to characterize dredged material before disposal to reduce the potentially adverse effect on the environment. Characterizing sediments as to the presence and concentration of contaminants in dredged material becomes increasingly more expensive as new contaminants of concern are added to the list of those whose presence must be assayed. Sediment evaluation is the main factor in making dredged material management decisions and is based on a "Management Strategy for Disposal of Dredged Material," as detailed in Francingues et al. (1985). This management strategy employs a "reason to believe" approach to sediment evaluation. The CE has made a commitment to this strategy as a management tool for dredged material disposal, including proper assessment of the environmental consequences.

210. Most of the dredged material, some 300 million cubic meters, removed from our Nation's waterways is uncontaminated or is acceptable for a wide range of disposal alternatives. Fortunately, the evaluation of this uncontaminated sediment does not require extensive testing and expense. There is a direct relationship between cost of sediment evaluation and number of contaminants and degree of contamination. Cost of sediment evaluation will increase with increased numbers of contaminants assayed and the degree of contamination. Generally, cost-reduction recommendations have the greatest potential for tangible cost saving when applied to contaminated sediment. Higgins (1988) provided recommendations to reduce costs and provide adequate environmental protection prior to disposal of dredged material. These recommendations include:

a. Proper design of the sampling plan, which includes

(1) Reviewing historical data.

(2) Using a scientifically based sediment sample collection scheme.

(3) Dividing project area into management units.

b. Proper collection and handling of sediment samples, including

(1) Collecting core samples.

(2) Storing sediment properly.

(3) Preparing composite samples.

c. Quality control and quality assurance as integral parts of sediment evaluation.

d. Use of chemical and biological screening techniques when appropriate.

e. Use of decision risk analysis to identify and correct weaknesses in the sediment evaluation process.

211. Due to the ever-present concern of reducing the cost of sediment characterization, a detailed discussion of each cost-reduction technique is presented in Appendix C.

Contractor data reporting

212. Data format. The data should be tabulated or displayed in a logical and understandable manner, with the appropriate number of significant figures and appropriate units, identified as to wet or dry weight basis. Concentration units should be milligrams per liter for water, milligrams per kilogram dry weight for sediment, and milligrams per kilogram either dry or wet weight for tissue samples, depending on the objectives of the project. Detection limits should be indicated for parameters where values are below the detection limit. Results of internal quality control checks on percent recovery of spikes and duplicate determinations of a single sample should be included in the report. The detailed discussion of significant figures, rounding off rules, statistical conventions, sample report forms, and laboratory data handling given in the "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (USEPA 1988) should be followed.

213. Reporting frequencies. Monthly progress reports summarizing all analytical and biological results are highly desirable. A summary of quality control/quality assurance activities and data should be submitted to the project manager by the contractor on at least a semiannual basis for long-term analytical service contracts and at quarterly intervals for projects of lesser

duration. The summary quality assurance reports should include precision and bias data collected from standard samples, reference material samples, and any split samples used in the contractor's internal quality control program. Where possible, these data are to be compared to expected values. Quality control charts may also be included in the summary reports as well as graphical presentations of precision and bias data. Friedman and Erdmann (1980) have detailed numerous methods of summarizing quality control data, one of which should be selected and followed in such reports.

214. Sample encoding and decoding. The practice of analytical laboratories assigning their own sequential numbers to all samples received for analysis is essential in a smoothly operating laboratory. It is mandatory, however, that the field station where the sample was taken be identified next to the sequential laboratory number. The field station identification should include sufficient information to allow precise placement of the sample in space and time independent of the contractor numbering system.

215. Quality control data review. The project manager should closely monitor routine data reports to ensure that replicates and spikes are being run. An onsite inspection is extremely helpful in ensuring that replicate and spiked sample analyses, as well as the entire internal quality control program of the contractor, are being carried out and correctly utilized. If the contractor is responsible for all phases of the study, the project manager should also ensure that the contractor is carrying out the spiking and splitting that would normally be part of the Government's quality assurance program.

216. It is difficult to provide firm guidance on how close replicate and split sample analyses should be or how close to 100 percent the recovery of sample spikes should be. The difficulty arises due to the statistical nature of the procedures required in the "Handbook for Analytical Quality Control" (USEPA 1988). In general, however, the majority of spiked samples should show 100 percent ± 10 percent recovery, and analyses of replicates and split samples should be within 15 to 20 percent of each other. When concentrations are near detection limits, the above goals may have to be relaxed somewhat.

217. The project manager should also closely examine the data for apparent decimal slippages, correct reporting of units, and identification of tissue analyses as either wet or dry weight basis. In general, the project

manager should pay close attention to detail when inspecting testing procedures and analytical data.

218. The quality, or lack thereof, of analytical data supplied by the contractor laboratory will become readily apparent from the samples submitted in the Government's quality assurance program. The project manager should closely monitor results of this program to ensure that problems are rapidly identified and corrected. Quality assurance data evaluation methods presented in the "Handbook for Analytical Quality Control" (USEPA 1988) and by Friedman and Erdmann (1980) should be followed.

219. Report milestones. The project manager should clearly specify in the scope of work the required frequency of progress reports and the time allowed to complete the project following the contract award. Project progress reports are normally required at monthly intervals. For analytical services contracts that may be in force for a number of years, sample turnaround time (time from sample receipt to analytical results report) should be specified.

220. Retention of samples and data. Unused parts of samples or sample extracts must be preserved by the best method available and stored for an appropriate time commensurate with the type of sample and the preservative used. Retention is required in the event that it becomes necessary to have some of each sample available for an unforeseen further analysis. Doubt as to retention of a specific sample should be resolved in favor of retention pending inquiry to the proper legal counsel regarding the specific case. In view of the possibility of legal action, raw data should be retained by the project manager for at least 7 years after the final report is received.

221. Disposal of contaminated sediment after testing. When deemed necessary, it is the responsibility of the contractor to dispose of all contaminated sediment used in testing. The contractor should therefore be prudent in his estimation of the volume of sediment required to conduct appropriate tests, because smaller volumes will facilitate disposal and reduce disposal cost. Disposal of all contaminated sediment should follow the guidelines outlined in the Resource Conservation and Recovery Act, 40 CFR 260-280 (42 USC 6901 et seq.); Toxic Substances Control Act, 40 CFR 702-799 (15 USC 2601 et seq.); Hazardous Materials Transportation Act, 49 CFR 172-177 (49 USC 1801 et seq.); and the Comprehensive Environmental Response, Compensation, and Liability Act, 40 CFR 300-306 (42 USC 9605 et seq.).

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APPENDIX A: EXAMPLE SCOPE OF WORK

STATEMENT OF WORK FOR
LONG-TERM AQUATIC DISPOSAL STUDY, DUWAMISH
WATERWAY, ELLIOTT BAY, SEATTLE, WASHINGTON

Background Information for Work To Be Done

1. The US Army Corps of Engineers was authorized by the River and Harbor Act of 1970 (Public Law 91-611) to conduct a comprehensive nationwide research study on the environmental effects of dredged material disposal. The task of developing and implementing the Dredged Material Research Program (DMRP) was assigned to the Environmental Effects Laboratory (EEL) of the US Army Engineer Waterways Experiment Station (WES), Vicksburg, MS.

2. The Environmental Impacts and Criteria Development Project of EEL has as one of its missions the identification and assessment of environmental impacts of the aquatic disposal of dredged material. Completed research in this project includes large-scale field investigations of ongoing Corps of Engineers dredging and disposal operations, the development of mathematical models to describe the movement and dispersion of dredged material, and laboratory studies of the effects of dredged material disposal on water quality and aquatic organisms. The DMRP as authorized is completed, and forthcoming results indicate the necessity of long-term (3 years or more) monitoring of select open-water disposal sites in order to ascertain the more subtle or chronic effects on the surrounding biota and physical nature of the area. One site selected for these studies is the Duwamish Waterway site in Seattle, WA.

3. The Duwamish Waterway study was unique in that it was the only estuarine research site and the only study of barge disposal among the five Aquatic Disposal Field Investigations. Postdisposal field research at this site was terminated in September 1976. Data collected during this study have been analyzed and are summarized in several WES reports. Examination of these data suggest that many research areas need to be assessed on a longer term basis. The one area that has not been adequately studied is the long-term (years) impact of open-water disposal on the estuarine environment, with particular emphasis on benthic macroinvertebrates.

Scope of Work

4. The work to be done hereunder consists of furnishing and delivering to WES all services, labor, material, supplies, and equipment necessary to conduct the Long-Term Aquatic Disposal Study, Duwamish Waterway, Elliott Bay, Seattle, WA.

Task I - Long-Term Disposal
Effects on Marine Benthic
Invertebrates at Disposal Area

5. Objectives. Task I objectives are as follows:

   a. The contractor will be required to measure the rates and patterns of benthic colonization at the disposal area and compare these rates with those of adjacent unimpacted areas over a 3-year period.

   b. The contractor shall estimate changes in diversity, species richness, and biomass at the disposal area and adjacent areas. In addition, the contractor shall estimate the relative value of the disposal area habitat in comparison with adjacent unimpacted areas over a 3-year period.

   c. The contractor shall examine physical and chemical changes in sediment texture of the upper few centimeters of the sediments and relate these changes to benthic activity and biological reworking processes.

6. Sampling design. Benthic invertebrate samples will be collected with a standard 0.1-m^2 Van Veen grab from a total of 20 stations in the study area. Three replicate grabs will be taken from each station two times per year. One additional sample will be taken from five of the above stations for benthic PCB uptake studies. Vessel positioning for each sample station shall be accurate to ±15 m.

7. Analysis. Each replicate benthic sample retained on a 1.0-mm screen will be sorted, enumerated, and identified to species level where possible. Sample volume will be determined in the field. Dry weight biomass (mg/m^2) will be determined for each sample. Benthic invertebrates data will be subjected to mutually agreeable parametric statistical comparisons to examine spatial and temporal trends and the interaction between sample stations and time. Samples collected for uptake studies will be sorted immediately for a select number of dominant benthic species from various trophic levels and

frozen immediately in tin foil. These samples will be analyzed for PCBs according to the procedure described by the US Environmental Protection Agency.*

Task II - Long-Term Stability of Dredged Material Deposit

8. Objectives. Task II objectives are as follows:

   a. The contractor will estimate temporal changes in size and shape of the dredged material deposit.

   b. The contractor will examine temporal sediment physical changes to include texture, composition, and dewatering.

   c. The contractor will investigate near-bottom current structure and suspended load at the disposal area and relate these to suspended and bed-load transport mechanisms.

9. Sampling design. High-resolution bathymetric surveys (200 kH_z) will be conducted at the disposal area twice a year for a period of 2 years by the US Government. These survey data will be supplied to the contractor, who in turn will reduce the data and produce bathymetric charts that will provide a basis for the determination of volumetric changes and movement of the dredged material. Replicated gravity cores (2 cores/cast) will be taken from each of 20 sample stations, twice per year. Each core will be used for grain size and other physical measurements such as percent water and void ratio. Bottom water current structure (speed and direction) will be measured in the disposal area two times per year (2 weeks before and 2 weeks following other sampling). Bottom transmissivity will be determined concurrently with the bottom currents. An appropriate number of bottom water samples will be taken to serve as calibration points for the transmissometer.

10. Analysis. The contractor will reduce bathymetric survey data from each cruise and will produce isopach maps of sediment thickness at the disposal site. Volumetric and areal changes in the disposal mound over time will be derived from these data. Grain size analysis will be performed on surface sediments according to the methods outlined by Folk.** Other physical measurements, such as porosity, void ratios, and density, will be taken by

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* J. F. Thompson, ed. 1974. "Analysis of Pesticide Residues in Human and Environmental Samples," Primate and Pesticide Laboratory, USEPA, Perrine, FL.

** R. L. Folk. 1968. "Petrology of Sedimentary Rocks," University of Texas, Austin.

commonly accepted methods. Current meter data and transmission data will be reduced by the contractor for each deployment period and correlated with sediment textural data to provide an estimate of the bottom stress and subsequent motion by bed-load and suspended load transport.

Task III - Long-Term
Mobilization of Polychlorinated
Biphenyls and Other Contaminants

11. Objectives. The contractor will measure the uptake of PCBs and other contaminants by select dominant benthic species of various trophic levels, and will relate body burdens of PCBs to PCB concentrations in sediments, interstitial water, and near-bottom water. The contractor will also examine the vertical distribution of PCBs and other contaminants in sediments and estimate the diffusion rate within the sediments and into the overlying water.

12. Sampling design. Five benthic stations will be sampled on a semi-annual basis to obtain sufficient animals for PCB uptake studies. Replicate sediment cores will be taken from each of these five stations for subsequent PCB and contaminant analyses on total sediment and interstitial water fractions. Near-bottom water will also be collected at each of these stations and filtered through 0.45-μ membrane filters.

13. Analysis. Sediment cores are to be extruded under a nitrogen atmosphere and sectioned into three fractions (from the top), each fraction being approximately 3 cm in thickness. Each section will be analyzed for pH, nitrate, phosphate, ammonia, total organic carbon, PCBs, sulfides, and oil and grease. Interstitial water samples will be analyzed for the same suite of properties. Near-bottom water samples will be analyzed for dissolved and particulate pH, PCBs, ammonium, nitrate, phosphate and silicate. Minimum analytical detection limits for PCBs in the following fractions will be required of the contractor: total 0.1 ppb, particulate 0.1 ppb, and dissolved 1 ppt.

Data Submission

14. All data will be submitted as soon as possible to the Government in a predetermined, mutually agreeable format in the form of a SAS (Statistical

Analysis System*) data set. The SAS data set must be transmitted to the Government on IBM 370-compatible magnetic tape, or other suitable media that must be agreed upon prior to transmittal. All transmitted data must be verified by the contractor, and a statement pertaining to the quality assurance must accompany the SAS data set.

Reports

Monthly progress reports

15. The contractor will be required to submit brief letter-type progress reports each month. The report will include copies of all raw data processed during the reporting period and presented in summary fashion, as well as a discussion of any significant observations and trends in the data.

Annual interim reports

16. The contractor will be required to prepare an interim report following each year's research which summarizes significant findings and observations, as well as recommendations for future research. This interim report will be submitted by the end of each fiscal year.

Final report

17. The final year of the study (FY 81) will result in the production of a final contract report that describes the results and conclusions of the entire study. This report will be prepared according to WES Instruction Report 0-76-1. Five copies of the draft final report will be submitted to WES for review. Upon return of the draft, the contractor will furnish WES a final photoreproducible copy of the report. Technical findings shall not be subject to approval of the Contracting Officer, but recommendations by the Contracting Officer for changes in the findings that are acceptable to the contractor will be incorporated in the final reproducible copy. Should such recommendations be unacceptable to the Contractor, the reproduction copy will contain an appropriate statement that the technical findings do not necessarily reflect the view nor have the concurrence of the US Army Corps of Engineers. Printing, binding, and distribution of the final report will be accomplished by the Government.

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* A. J. Barr et al. 1976. "A User's Guide to SAS 76," SAS Institute, Raleigh, NC.

APPENDIX B: CHAIN-OF-CUSTODY PROCEDURES

CHAIN-OF-CUSTODY PROCEDURES*

1. The following procedures are a modification of the Chain-of-Custody Procedures for Microbiological Samples contained in the Handbook for Analytical Quality Control in Water and Wastewater Laboratories.**

2. A sample may be considered in a person's "possession" or "custody" if

   a. It is in any person's actual physical possession.

   b. It is in his view, after being in his physical possession and before any transfer.

   c. It is under lock and key, while not in actual physical possession, in a manner that it could not be tampered with, and that person has access to a key.

3. Samples must be kept in such a manner that chemical composition cannot be changed. A sample custodian must keep sample(s) in hand or view until there is a seal on the container unless it is secured in an area of limited access. In cases where sample data may be used for litigation, it is not enough to have the sample in a locked building. A custodial record must be maintained in a bound book specifying each location in which a sample was stored and the period of storage.

Filling Out the Sample Label

4. Immediately after the sample has been collected, the Field Crew Chief (FCC) must fill out the "Chain-of-Custody Record," which is a tagged label that describes the sample and lists the people who have had custody of it. A sample tag or label must be attached to each sample container. The sample tag or label should be filled out as follows:

NOTE: ALL ENTRIES MUST BE LEGIBLE

   a. SAMPLE NUMBER: Record in field log as well as on tag.

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* This appendix was prepared by the Analytical Laboratory Group, Environmental Laboratory, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

** Report EPA 600/4-79-019 (1979), Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, OH [3rd ed., 1988].

b. SOURCE OF SAMPLE: Be specific.

c. PRESERVATIVE: Be specific on reagent and volume.

d. SAMPLE COLLECTOR/WITNESS: Signatures only.

e. REMARKS: Specify; analyses to be performed; whether sample is grab or composite; for composite sample, specify the type of composite, e.g. 24-hr composite, middepth-bottom composite, composite of three sediment samples, etc.; specify unusual characteristics that may require special laboratory handling, e.g., nauseous odor, etc.

f. RECEIPT OF SAMPLE (on reverse of tag or bottle label): This is to be filled in by each person who receives the sample after it leaves the sampler. However, only a person who has actual physical access to a sample needs to sign for the sample.

g. DISPATCH OF SAMPLE (on reverse of tag or on the label): This is to be filled in only by a courier who takes the sample from the FCC and ships it. Be sure to retain the shipping receipt.

Sealing the Samples

5. Before the samples leave the possession of the FCC, the FCC must place a seal on the sample container. This seal must be of a type and be placed on the sample container such that nothing can be added to or taken from the sample container without breaking the seal. To prevent the removal of the seal and its replacement by someone else, the seal must have the following written on it:

a. The date the sample(s) were sealed. This should be the same date that the sample(s) were collected.

b. The number assigned to the sample or inclusive numbers of samples contained in the sample container.

c. The signature (legible) of the sampler.

6. For samples being shipped in an ice chest, an adhesive label containing the proper information may be used, preferably in conjunction with a lock, to seal the container. For a single sample that will not be placed in a container, tape containing the proper information can be used as a seal.

Notebook Procedure for Persons Other Than the FCC

7. Each individual who has custody of samples must keep a bound notebook in which he records information on receipt and disposition of samples.

This is equally as important for an analyst as it is for the sampler. For a chemist or analyst, entries must include the following information:

a. The date and time the sample was received.

b. The number assigned to the sample.

c. The name of the individual or the shipping agency from whom the sample was received.

d. If received from a shipping agency, the number of the Government Bill of Lading, certified mail or UPS receipt, or any other identification on the package.

e. What the recipient did with the sample, from the time that he received it until it left his custody. To the extent that the following are applicable, they should be recorded in the notebook.

(1) Exactly where the sample was stored and the times that it was stored there. (It must be kept in a locked room, cabinet, or other container, not just in a locked building.)

(2) To whom the sample was given. The name of the individual must be recorded, even if the recipient did not sign the sample tag.

Sample Shipment and Delivery

8. The minimum possible number of individuals should handle a sample. The reason for this is that the more people who handle it, the more chance there is for a "break" in the Chain-of-Custody.

9. When possible, the individual who collected and sealed the sample should package the sample and deliver the tagged and sealed sample to the shipping agency. If this is not practical, then at a minimum the sampler must fill out the sample tag and attach it to the sample, seal the sample, and deliver the tagged and sealed sample to a courier to package for shipment. The courier must then sign the sample tag as having received it from the sampler, package the sample, and deliver it to the shipping agency or hand-carry it to the laboratory. More than one courier should never be used.

10. At the time a sample is shipped, the sampler (or other custodian) should notify by telephone the address to expect the sample(s) and, in addition, to relay a list of the contents of each package. If the package is shipped on a Government Bill of Lading, the Bill of Lading should list the contents of the package.

Laboratory Custody Procedures

11. Suitable laboratory procedures during custody of samples include the following:

a. The laboratory shall designate a sample custodian and an alternate custodian to act in his absence. In addition, the laboratory shall set aside a sample storage security area. This should be a room with refrigerator space that can be securely locked from the outside.

b. Samples should be handled by the minimum possible number of persons.

c. Incoming samples shall be received only by the custodian, who will indicate receipt by signing the chain-of-custody record sheet accompanying the samples and retaining the sheet as a permanent record. Couriers picking up samples at the airport or post office shall sign jointly with the laboratory custodian. Samples should not be accepted if the seal is broken. The Field Sampling Crew Chief should be notified immediately.

d. Upon receipt of acceptable samples, the custodian should place the samples in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.

e. Only the custodian should distribute samples to personnel who are to perform tests.

f. The analyst's records should be kept in a laboratory notebook or on an analytical worksheet, identifying samples and results of the testing. The notes must be dated specifying the analyst performing each test, and should include an observation of abnormalities that occurred during the testing procedure. The notes must be retained as a permanent record in the laboratory. In the event the data are introduced into a litigation and the person who performed the tests is not available as a witness at the time of trial, the Government may introduce the notes in evidence under the Federal Business Records Act.

g. Methods of laboratory analyses must be used as required in the scope of work.

h. Laboratory personnel are responsible for the care and custody of a sample once it is handed to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were completed.

i. Once the sample analyses are completed, the unused portion of the sample, together with identifying labels and other documentation, must be returned to the custodian. The returned, tagged sample should be retained in the custody room until permission to destroy the sample is received by the custodian.

j. Samples should be destroyed only upon the order of the District Engineer, in consultation with legal counsel, or when it is certain that the information is no longer required. The same destruction procedure is true for tags, labels, and laboratory records.

APPENDIX C: COST-REDUCTION TECHNIQUES

COST-REDUCTION TECHNIQUES*

Design of the Sampling Plan

Review of historical data

1. Historical information is very important in the design of a cost-effective sediment sampling plan. Review of historical data gives the sampling plan designer the opportunity to apply the "reason to believe" rationale. A key to the value of historical data is the adequacy and accuracy of the documentation attached to it. To be of value, historical data should provide the reviewer with the date and exact location of the sample, how it was collected, and how it was handled or stored. Historical data lacking detailed information may not provide an accurate representation of the waterway to be dredged. Use of incomplete historical data may adversely impact the design of the sampling plan. Historical data are considered to remain valid up to 2 years.

Selection of sample collection sites

2. Appropriate historical data can be applied to provide a presampling characterization of the dredging project and can assist the sampling plan designer in selecting the best method. The sampling methods most often used to characterize sediments are (a) haphazard, (b) worst-case, (c) random, (d) stratified random, and (e) exhaustive.

3. The haphazard method is not based on sound scientific principles. It is based on the sampling plan designer's personal biases or is used to satisfy the concerns of special interest groups. This method should not be employed on CE projects and should be discouraged on non-Federally funded dredging projects.

4. The worst-case method is considered to be low cost. This technique concentrates on specific areas that were identified as contaminated (referred to as "hot spots") through historical data. Incomplete sediment characterization in the project area is an inherent problem when this method is used. More complete characterization of the project area may later be required by

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* This appendix was prepared by Todd Higgins, Environmental Laboratory, US Army Environmental Waterways Experiment Station, Vicksburg, MS.

other regulatory agencies, thus requiring the collection of more samples and increasing the cost.

5. The random sampling method is most useful when no reliable historical data are available or when available information indicates that sediment within the area is homogeneous. In this case, properly employed random sampling will result in high confidence in the characterization of the sediment.

6. The stratified random sampling method is similar to the random method in that the entire project area is divided into units and sampled. With historical data, sampling can be conducted in units where contamination is most likely to be found. This method is scientifically sound and permits sediment zones to be characterized with a high degree of confidence.

7. In the exhaustive sampling method the project is divided into equal-sized units, and each unit is sampled. This method is not recommended for routine sampling due to the high cost. It does permit characterization of the sediment with a high degree of confidence; however, it is more useful in projects that have several sources that contribute to contamination.

Management units

8. Management units are areal or volumetric subdivisions of the dredging project designed to enhance management of the sediment sampling and dredging programs. The major cost-saving benefit from dividing project areas into management units is that each management unit can be characterized independently. Consequently, management units can be managed either individually or collectively, thereby reducing the volume of sediment disposed of in confined disposal sites at higher cost.

Collection of Sediment Samples

Sediment sample collection

9. The sampling plan designer should keep a broad view of the cost of the sample collection operation when selecting sampling sites and determining the number of samples to be collected. Normally, the cost of collecting, handling, transporting, and storing additional samples is minimal when compared to the total cost of the sample collection effort. Therefore, the sampling plan designer should take additional samples in areas where potential contamination may exist and store them for further analysis should it be required.

By collecting and storing additional samples on the initial effort, the need for a follow-up sample collection effort may be avoided.

Storage techniques

10. The most important aspect of sample storage is that it will potentially reduce the need to resample a project site, thereby reducing or eliminating the cost of resampling. Preservation techniques are discussed in paragraphs 170-187 of the main text. Required sediment containers, holding times, and preservation techniques for the different parameters are discussed in Appendix D.

Sample compositing

11. The major cost of characterizing a sediment has to do with the number of samples, not the number of parameters for which a sediment is assayed. Therefore, compositing and homogenating several samples into one for analysis may result in significant cost savings. A carefully planned compositing scheme can reduce costs and improve the confidence level in the data by reducing variability.

Quality control/quality assurance

12. The benefits of a good quality control program are many, but the two most important benefits are increased confidence in management decisions and decreased program costs. Increased confidence develops from having a scientifically sound basis for collecting samples, using the best collection method, handling and storing samples properly, and having confidence that the analytical lab performed the analysis correctly. Cost savings are achieved by eliminating resampling, reducing reanalysis, and characterizing the sediment in a manner that permits individual management units to be disposed of in an appropriate manner.

Chemical and Biological Screening

Chemical screens

13. The cost for organic analyses can be monumental; for example, the cost for polychlorinated biphenyl (PCB) analysis is $175 (1988). Depending on the number of sampling sites, the number of replicates per site, and the samples per profile, it is apparent that great expense can be expended on any one parameter. The use of screening assays may be appropriate when organic contaminant concentrations are of concern. The screens may eliminate the need to

analyze for certain organics and, furthermore, may reduce the cost of dredged material disposal if the screens do not indicate the presence of contaminants of concern.

Biological assessments

14. No technically defensible cost-reduction techniques are currently available for regulatory biological assessment tests. Biological screens that are available may be useful in comparing and ranking sediments within a project area. Daphnia, mysid, and amphipod sediment toxicity tests are considered to be screening tests. Mysid shrimp have also been used as an internal standard and as the basis for quality assurance. Biological screens are useful in determining where to concentrate more intensive and expensive studies.

Dredged Material Disposal Decision Risk Analysis

15. Risk analysis is the estimated numerical value that depicts the confidence level in a management decision. Risk analysis can improve sediment evaluation, by identifying weak points in the decision-making process, and can allow for corrective measures to the instituted. The risk analysis will also serve as an educational tool, allowing weaknesses to be identified, analyzed, and hopefully prevented in the future.

APPENDIX D: RECOMMENDED PROCEDURES FOR SAMPLE COLLECTION, CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Analysis or Test	Collection Method	Amount Required	Container	Preservation Technique	Storage Conditions	Storage Duration
			Sediment			
Chemical/physical analyses						
Bulk analysis	Dredge/grab/corer	1-2 ℓ	Plastic jar/bag; glass with Teflon-lined lid for organics	Completely fill container and refrigerate	4° C/dark, airtight	6 months
Trace metals	Dredge/grab/corer	200 ml	Acid-rinsed plastic or glass with Teflon-lined lid	If analyzed within 24 hr refrigerate; if >24 hr, freeze	4° C ≤ 24 hr; -20° C > 24 hr	6 months
Trace organics (PCBs, pesticides, heavy molecular weight hydrocarbons)	Dredge/grab/corer	1,000 ml	Solvent-rinsed glass container, Teflon-lined lid	If analyzed within 24 hr refrigerate; if >24 hr, freeze	4° C ≤ 24 hr; -20 C° > 24 hr	7 days
Particle size	Dredge/grab/corer	3-25 g	Plastic or glass	Lugols solution and refrigerate	4° C	Indefinite
Elutriate test	Grab/corer	3 ℓ	Plastic or glass; if organics analysis, glass with Teflon-lined lid	Completely fill and refrigerate	4° C/dark, airtight	1 week
Sediment fractionation	Grab/corer	1 ℓ	Glass with Teflon-lined lid	Completely fill and refrigerate	4° C/dark, airtight	1 week

(Continued)

Analysis or Test	Collection Method	Amount Required	Container	Preservation Technique	Storage Conditions	Storage Duration
Biological tests						
Dredged material	Corer/grab	12-15 ℓ per sample	Plastic bag or container	Completely fill and refrigerate; sieve	4° C/dark, airtight	6 weeks
Reference sediments	Grab/corer	45-50 ℓ per test	Plastic bag or container	Completely fill and refrigerate; sieve	4° C/dark, airtight	6 weeks
Control sediments	Grab/corer	21-25 ℓ per test	Plastic bag or container	Completely fill and refrigerate; sieve	4° C/dark, airtight	6 weeks
			Water			
Particulate analysis	Discrete sampler or pump	500-2,000 ml	Plastic or glass	1-mm sieve Lugols solution and refrigerate	4° C	Indefinite
Trace metals analysis	Discrete sampler or pump	100-2,000 ml	Acid-rinsed plastic or glass with Teflon-lined lid	HNO_3 to pH <2	Ambient	6 months
Mercury	Discrete sampler or pump	500 ml	Glass	HNO_3 to pH <2	Ambient	2 weeks
Selenium	Discrete sampler or pump	500 ml	Plastic or glass	HNO_3 to pH <2	Ambient	90 days
Nitrogen	Discrete sampler or pump	100-200 ml	Plastic or glass	H_2SO_4 to pH <2 refrigerate	4° C	24 hr

(Continued)

Analysis or Test	Collection Method	Amount Required	Container	Preservation Technique	Storage Conditions	Storage Duration
Organophosphates and carbonates	Discrete sampler or pump	1 ℓ	Glass	H_2SO_4 to pH <3; 10 g Na_2SO_4	Ambient	Indefinite
Chlorinated phenoxy acid herbicides		1 ℓ	Glass	H_2SO_4 to pH <2; refrigerate	4° C	Indefinite
Oil and grease		200 ℓ	Glass	H_2SO_4/HCl to pH <2; refrigerate	4° C	24 hr
Chemical oxygen demand		200 ℓ	Plastic or glass	H_2SO_4 to pH <2; refrigerate	4° C	7 days
Total organic carbon		100 ml	Plastic or glass	H_2SO_4 to pH <2; refrigerate	4° C	<48 hr
Total inorganic carbon		100 ml	Plastic or glass	Airtight seal; refrigerate	4°C	Indefinite
PCBs, organo-chlorine pesticides		1-2 ℓ	Glass	Refrigerate	4° C	Indefinite
Phenolics		1 ℓ	Glass	0.1-1.0 g $CuSO_4$ H_3PO_4 to pH <4; refrigerate	4° C	24 hr
Chlorine demand		--	Plastic or glass	None	Immediate analysis	Immediate analysis

(Continued)

Analysis or Test	Collection Method	Amount Required	Container	Preservation Technique	Storage Conditions	Storage Duration
Soluble reactive phosphates	Discrete sampler or pump	--	Plastic or glass	Filter, refrigerate	4° C	24 hr
Total phosphorus		--	Plastic or glass	Refrigerate	4° C	7 days
Redox potential		100 ml	Plastic or glass	Refrigerate	4° C	6 hr
Total solids		200 ml	Plastic or glass	Refrigerate	4° C	7 days
Volatile solids		200 ml	Plastic or glass	Refrigerate	4° C	7 days
Sulfides		--	Plastic or glass	2 ml ZnOAc	Ambient	24 hr
Trace metals	Trawl/Teflon-coated grab	30 g	Plastic bag	Handle with nonmetallic forceps, plastic gloves; quick freeze	-20° C	Indefinite
PCBs and chlorinated pesticides	Trawl/Teflon-coated grab	100 g	Hexane-rinsed aluminum foil	Handle with hexane-rinsed stainless steel forceps; quick freeze	-20° C	Indefinite
Polychlorinated aromatic hydrocarbons	Trawl/Teflon-coated grab	50 g	Hexane-rinsed aluminum foil	Handle with hexane-rinsed stainless steel forceps; quick freeze	-20° C	Indefinite

APPENDIX E: ADDITIONAL REFERENCES ON BIOLOGICAL TESTING

ADDITIONAL REFERENCES ON BIOLOGICAL TESTING

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APPENDIX F: ESTIMATION OF VOLUME OF WATER REQUIRED FOR CONDUCTING ONE SOLID PHASE BIOASSAY

ESTIMATION OF VOLUME OF WATER REQUIRED FOR
CONDUCTING ONE SOLID PHASE BIOASSAY

Assumptions

1. Reference and control sediments are not the same; therefore, at least three treatments will be established:

   a. Dredged material under investigation. (This example includes dredged material from only one sampling site. In practice, several sampling sites would probably be included, and the number of treatments in the calculations increased accordingly.)

   b. Reference sediment.

   c. Control sediment.

2. Three species will be tested. Two of these are compatible and may be tested together in the same aquaria. The third is not compatible with the others and must be tested separately. All species will be tested concurrently.

3. Five replicates of each treatment will be established.

4. The volume of each aquarium is 37.8 ℓ.

5. The rate of flow will be such that a volume equal to six times the aquarium volume will be added to each aquarium over the course of 1 day.

6. The actual length of time the water flows through the aquaria will be 12 days: 2-day acclimation period followed by a 10-day test period.

Calculations

7. The number of aquaria required will be

$$2 \text{ aquaria} \times 3 \text{ treatments} \times 5 \text{ replicates} = 30$$

8. The volume each aquarium will receive in 1 day will be

$$6 \times 37.8 \text{ ℓ} = 226 \text{ ℓ}$$

equivalent to a continuous flow rate of 160 ml/min.

9. The total volume of water required in 12 days will be

$$12 \times 30 \times 226 \text{ ℓ} = 81{,}360 \text{ ℓ} = 21{,}495 \text{ gal}$$

APPENDIX G: ESTIMATION OF SEDIMENT VOLUMES REQUIRED FOR CONDUCTING ONE COMPLETE DREDGED MATERIAL BIOASSAY

ESTIMATION OF SEDIMENT VOLUMES REQUIRED FOR CONDUCTING ONE COMPLETE DREDGED MATERIAL BIOASSAY

Solid Phase Test

Assumptions

1. Reference and control sediments are not the same; therefore, three treatments will be established:

   a. Dredged material under investigation. (This example includes dredged material from only one sampling site. In practice, several sampling sites would probably be included and the number of treatments in the calculations increased accordingly.)

   b. Reference sediment.

   c. Control sediment.

2. Three species will be tested. Two of these are compatible and may be tested together. The third is not compatible with the others and must be tested separately. All species will be tested concurrently.

3. Five replicates (REPS) of each treatment will be established.

4. Standard 10-gal glass aquaria having bottom measurements of 26 × 51 cm will be used.

5. A 30-mm layer of the reference sediment will be established on the bottom of all aquaria in treatments 1a and 1b, and a 30-mm layer of control sediment will be established on the bottom of all aquaria in treatment 1c.

6. After an initial 2-day acclimation period, a 15-mm layer of dredged material will be added to the 10 aquaria in treatment 1a; a 15-mm layer of reference material will be added to the 10 aquaria in treatment 1b; and a 15-mm layer of control sediment will be added to the remaining 10 aquaria.

Calculations

7. The total volume of reference sediment required will be

$$(26\ \text{cm} \times 51\ \text{cm} \times 30\ \text{mm} \times 5\ \text{REPS} \times 2\ \text{treats} \times 2\ \text{aquaria}) = (26\ \text{cm} \times 51\ \text{cm} \times 15\ \text{mm} \times 5\ \text{REPS} \times 2\ \text{aquaria} \times 1{,}000^{-1} = 99.5\ \ell = 26.2\ \text{gal}$$

8. The total volume of dredged sediment will be

$$(26\ \text{cm} \times 51\ \text{cm} \times 15\ \text{mm} \times 5\ \text{REPS} \times 2\ \text{aquaria}) \times 1{,}000^{-1} = 19.9\ \ell = 5.2\ \text{gal}$$

9. The total volume of control sediment will be

$$(26\ \text{cm} \times 51\ \text{cm} \times 45\ \text{mm} \times 5\ \text{REPS} \times 2\ \text{aquaria}) \times 1{,}000^{-1} = 59.7\ \ell = 15.7\ \text{gal}$$

Liquid Phase Test and Suspended Particulate Phase Test, Combined Total Volumes of Sediment for One Complete Bioassay

Assumptions

10. A fish, a mysid shrimp, a zooplankter, and an algal species will be tested.

a. In each of these tests for each species, there will be three replicates of each treatment.

b. There will be three treatments in each test.

c. The treatments will consist of 100, 50, and 10 percent liquid or suspended particulate phase.

d. The portion of each volume in each container represented by the dredged sediment will be one fifth of the total.

e. The fish will be tested in both phases, and each container volume will be 37.8 ℓ.

f. The mysids will be tested in both phases, and the volume in each container will be 2.0 ℓ.

g. The algae will be tested in one phase, and the volume of each container will be 500 ml.

h. The zooplankton will be tested in one phase, and the volume of each container will be 200 ml.

Calculations

11. The combined total volumes of dredged material required to conduct one complete liquid phase and one complete suspended particulate dredged sediment bioassay are as follows:

a. Volumes required for fish

$$\left(\frac{37.8\ \ell \times 0.2}{0.8} + \frac{37.8\ \ell \times 0.1}{0.9} + \frac{37.8 \times 0.02}{0.98}\right) \times 3\ \text{REPS} \times 2\ \text{phases} = 86.5$$

b. Volumes required for mysids

$$\left(\frac{2.0\ \ell \times 0.2}{0.8} + \frac{2.0\ \ell \times 0.2}{0.9} + \frac{2.0\ \ell \times 0.02}{0.98}\right) \times 3\ \text{REPS} \times 2\ \text{phases} = 4.58$$

c. Volumes required for algae

$$\frac{0.5\ \ell \times 0.2}{0.8} + \frac{0.5\ \ell \times 0.1}{0.9} + \frac{0.5\ \ell \times 0.02}{0.98}$$

$$\times 3\ \text{REPS} \times 1\ \text{phase} = 0.57$$

d. Volumes required for zooplankton

$$\frac{0.2\ \ell \times 0.2}{0.8} + \frac{0.2\ \ell \times 0.1}{0.9} + \frac{0.2\ \ell \times 0.02}{0.98}$$

$$\times 3\ \text{REPS} \times 1\ \text{phase} = 0.23$$

e. Total = 91.9 ℓ or 24.2 gal

12. The total volume of dredged material required is then

$$5.2 + 24.2 = 29.4\ \text{gal}$$

13. Because of inevitable loss during handling and preparation, these calculated volumes should be given a generous loss factor. If 15 percent loss is assumed, the requirements become

a. Reference sediment: 26.2 + 3.9 = 30.1 gal

b. Dredged sediment: 29.4 + 4.4 = 33.8 gal

c. Control sediment: 15.7 + 2.4 = 18.1 gal

APPENDIX H: GENERAL GUIDELINES FOR HANDLING CONTAMINATED SEDIMENT

GENERAL GUIDELINES FOR HANDLING CONTAMINATED SEDIMENT

1. Due to the increased industrial discharge of waste into our Nation's waterways, sediment contamination is increasing at an alarming rate. As a result, the Corps is being asked to handle more and more sediments with various contaminants at different levels of concentrations. Sediment samples may contain heavy metals, hydrocarbon compounds, and pathogens from untreated sewage. To protect laboratory and field personnel, safety procedures must be developed and followed when handling contaminated sediment.

General Requirements for Personnel Safety

2. Precautions to be observed by persons who handle contaminated sediment are given below.

a. Before handling contaminated sediment, all individuals should be required to attend a safety presentation on the handling of hazardous material.

b. All individuals handling contaminated sediment should wear protective clothing commensurate with the level or type of contamination.

c. All individuals handling contaminated sediment should do so only in facilities designated for that material. These sediments are not to be handled in areas where unauthorized personnel will come in contact with them.

d. Personnel may be asked to submit to blood and urine tests before handling contaminated sediments, routinely during the project, and after sediment testing. These blood and urine tests are intended as a monitoring procedure to ensure the safety of the individual handling the sediment.

e. Raw sewage carries a number of pathogenic microorganisms. It is suspected that some sediment may contain one or more pathogenic bacteria, including shigella, leptospira, vibrio (causes cholera), and mycobacterium (causes tuberculosis and leprosy), along with various types of pathogenic viruses. Individuals handling sediments with untreated sewage must be careful to follow the established safety guidelines.

Exposure Pathways

Ingestion

3. Toxic and pathogenic materials will produce undesirable results if ingested. Ingestion may occur from a splash of material striking the mouth or

lips, or indirectly, by ingestion of materials that have come in direct contact with the sediment sample (i.e., by eating, smoking, or drinking with dirty hands). The eyes and nose are also potential pathways.

Inhalation

4. Contaminants can enter the body and produce harmful results when vapors, aerosol dusts, or aerosol droplets are inhaled. This process is particularly important during mixing and sediment loading procedures. In most cases, only organic compounds are sufficiently volatile to produce significant exposure through inhalation of vapors. Therefore, laboratory personnel may be exposed to inorganic and organic chemicals and to pathogens through inhalation of aerosol dusts and droplets.

Transdermal transport

5. A major pathway for pathogens to enter the body is through breaks in skin, cuts, burns, or abrasions. Intact skin is an effective barrier to most pathogens and dilute inorganic chemicals. However, many organic chemicals can be readily absorbed into the body through the skin.

Protective Equipment

6. Recommended items of protective clothing are listed below and described in the following paragraphs.

a. Full-face chemical cartridge respirator (with an organic chemical cartridge and dust filter).

b. Pressure-demand airline respirator.

c. Full face shield.

d. Polyethylaminated or saran-coated tyvek disposable coverall.

e. Neoprene gauntlet gloves.

f. PVC disposable gloves.

g. Neoprene rubber boots.

h. Surgical scrubs.

7. To prevent ingestion of contaminated sediment, a face shield should be worn during sediment mixing, pouring, or handling in any manner that may result in splashing.

8. Appropriate protection against exposure by inhalation will vary according to working conditions and the sediment involved. For example, if the sample is handled in a well-ventilated area and no aerosol particles are

being generated, a full-face chemical cartridge respirator is recommended. However, in a poorly ventilated area where vapors and aerosol dust have a propensity to accumulate, a pressure-demand airline respirator is recommended.

9. Skin contact with contaminated sediment should be avoided by wearing suitable impervious protective clothing (saran or polyaminated tyvek coveralls). Tyvek coveralls (saran or polyaminated) with elasticated wrists are recommended and should be worn. The elasticated ankles should be worn over the top of neoprene boots so that any splashes will not dribble down inside footwear. Two pairs of gloves should be worn--inner and outer. The inner gloves should be disposable PVC gloves and should be worn under the sleeves of the tyvek coveralls. The outer gloves should be neoprene gauntlet gloves and should be worn over the sleeves of the tyvek coveralls.

10. Polyethylene sheeting should be placed under all test and mixing apparatus as a contamination preventive measure. This polyethylene sheeting will prevent needless contact with the laboratory surface and make cleanup easier.

Cleanup

11. Cleanup is an essential part of a safe laboratory environment. The procedure is as follows:

a. Contaminated sediment should be removed from all equipment using machine wipes. Used wipes are considered hazardous and should be disposed of in the same manner as coveralls (see below).

b. All equipment is rinsed in the laboratory sink after cleaning. The sink is then thoroughly cleaned.

c. The polyethylene sheeting is disposed of in a disposal drum.

d. Lids are fastened securely on the drums.

e. Coveralls (used as protective clothing) and surgical scrubs (worn underneath the coverall rather than personal clothing) are removed and placed in a disposal drum.

f. The disposal drum is labeled and disposed of according to the Hazardous Materials Transportation Act (49 CFR 172-177, as amended, 49 U.S.C. 1801, et seq.).

APPENDIX I: STANDARD REFERENCE MATERIALS FOR TRACE INORGANIC AND ORGANIC CONSTITUENTS

Trace Elements

Element**	Standard Reference Material* 1643b Water 950 ml	1646 Estuarine Sediment 75 g	2704 Buffalo River Sediment 50 g	1577a Bovine Liver 50 g
Aluminum		6.25%	6.11	(2)
Antimony		(0.4)	3.79	(0.003)
Arsenic	(49)† ng/g	11.6	23.40	0.047
Barium	44 ng/g		414.00	
Beryllium	19 ng/g	(1.5)	--	
Bismuth	(11) ng/g		--	
Bromine	B(94) ng/g		--	(9)
Cadmium	20 ng/g	0.36	3.45	0.44
Calcium		0.83%	2.60	120.00
Carbon			3.35	
Cerium		(80)	--	
Cesium		(3.7)	--	
Chlorine			--	0.28%
Chromium	18.6 ng/g	76	135.00	
Cobalt	26 ng/g	10.5	14.00	0.21
Copper	21.9 ng/g	18	99.00	158.00
Europium		(1.5)		
Gallium				
Germanium		(1.4)		
Hafnium				
Hydrogen				
Indium				
Iodine				
Iron (total)	99 ng/g	3.35%	4.11	194.00
Lanthanum	23.7 ng/g			

(Continued)

* Description of SRMs includes designation number, type, and unit size.
** Nominal concentrations, in micrograms per gram unless otherwise noted.
† Values in parentheses are not certified, but are given for information only.

Element	Standard Reference Material*			
	1643b Water 950 ml	1646 Estuarine Sediment 75 g	2704 Buffalo River Sediment 50 g	1577a Bovine Liver 50 g
Lead		28.2	161.00	0.135
Lithium		(49)		
Magnesium		1.09%		600.00
Manganese	28 ng/g	375	555.00	9.90
Mercury		0.063	1.44	0.004
Molybdenum	85 ng/g	(2.0)	--	3.5
Nickel	49 ng/g	32	44.10	
Nitrogen			--	10.7%
Phosphorus		0.054%	0.0998	1.11%
Potassium		(1.4%)	2.00	0.996%
Rubidium		(87)		12.5
Samarium				
Scandium		(10.8)		0.71
Selenium	9.7 ng/g	(0.6)		
Silicon		(31%)	29.07	0.243%
Silver	9.8 ng/g		--	
Sodium		(2.0%)	0.547%	0.138
Strontium	227 ng/g			0.78%
Sulfur		(0.96%)		
Tellurium		(0.5)		
Thallium	8.0 ng/g	(0.5)	1.21	(0.003)
Thorium		(10)	--	
Titanium		(0.51%)	0.458	
Tungsten			--	
Uranium			3.13	0.00071
Vanadium	45.2 ng/g	94	95.00	0.099
Zinc	66 ng/g	138	438.00	123.00

Organic Constituents

Constituent	Standard Reference Material 1580 µg/g	1582 µg/g	1644 µg/kg	1647 µg/ml	1649 µg/g	1650 µg/g
Anthracene			16.6 to 60.1	3.29		
Benz[a]anthracene		3.0	3.38 to 12.8	5.03	2.6	6.5
Benzo[a]pyrene	21	1.1	0.59 to 2.26	5.30	2.9	1.2
Benzo[e]pyrene	18					(10)*
Fluroanthene	54	2.5		10.1	7.1	51
o-Cresol	385					
Phenol	407					
Perylene	3.4	31				
Pyrene	104			9.84		(0.13)
2,6-Dimethylphenol	175					48
Benzo[f]quinoline (5,6-Benzoquinoline)	16					
Naphthalene				22.5		
Acenaphthylene				19.1		
Acenaphthene				21.0		
1-Nitropyrene						19
Fluorene				4.92		(71)
Phenanthrene		101		5.06		(22)
Chrysene				4.68		
Benzo[b]fluoranthene				5.11		
Benzo[k]fluoranthene				5.02		(2.1)
Benzo[ghi]perylene				4.01	4.5	2.4
Dibenz[a,h]anthracene				3.68		
Indeno[1,2,3-cd]pyrene				4.06	3.3	(0.23)
Dibenzothiophene		33				

* Values in parentheses are not certified, but are given for information only.

SRM 1639 - Certified Concentration of Halocarbons at 23° ± 3° C

Compound	Concentration, ng/µl
Chloroform	6,235
Chlorodibromomethane	124.6
Bromodichloromethane	389.9
Bromoform	86.5
Carbon tetrachloride	157.0
Trichloroethylene	85.8
Tetrachloroethylene	40.6

SRM 1581 - Polychlorinated Biphenyls in Oils

Matrix	Aroclor Type	Concentration, µg/g
Motor oil	1242	100
Motor oil	1260	100
Transformer oil	1242	100
Transformer oil	1260	100

SRM 1583 - Chlorinated Pesticides in 2,2,4-Trimethylpentane

Pesticide	Concentration	
	µg/g	µg/ml, 23° C
Y-BHC (Lindane)	1.11	0.77
d-BHC	0.76	0.53
Aldrin	0.86	0.59
Heptachlor epoxide	(0.997)*	
4,4'-DDE (p,p'-DDE)	1.23	0.85
4,4'-DDT (p,p'-DDT)	1.90	1.31

* Value in parentheses is not certified, but is given for information only.

SRM 1584 - Priority Pollutant Phenols in Methanol

Compound	Concentration, µg/ml, 23° C
2-Chlorophenol	64.4
Phenol	29.7
2-Nitrophenol	25.2
2,4-Dimethylphenol	51.6
2,4-Dichlorophenol	35.6
4-Chloro-m-cresol	27.4
2,4,6-Trichlorophenol	20.4
4-Nitrophenol	20.7
4,6-Dinitro-o-cresol	20.1
Pentachlorophenol	15.4
2,4-Dinitrophenol	(22.4)

SRM 1586 - Isotopically Labeled and Unlabeled Priority Pollutants in Methanol

Compound	Concentration, µg/g	
	1586-1 (unlabeled)	1586-2 (labeled)
Carbon tetrachloride	128.5	124.4
Benzene	101.1	99.0
Chlorobenzene	133.0	144.0
Phenol	117.0	116.0
Nitrobenzene	126.0	134.5
2-Nitrophenol	103.6	101.9
2,4-Dichlorophenol	102.5	82.2
Naphthalene	126.5	126.6
Bis(2-ethylhexyl)phthalate	63.9	60.4
Benzo[a]pyrene	49.2	44.1

SRM 1587 - Nitrated Polycyclic Aromatic Hydrocarbons in Methanol

Compound	Concentration	
	µg/g	µg/ml, 23° C
2-Nitrofluorene	9.67	7.64
9-Nitroanthracene	5.01	3.96
3-Nitrofluoranthene	9.24	7.30
1-Nitropyrene	8.95	7.07
7-Nitrobenz[a]anthracene	9.27	7.32
6-Nitrochrysene	8.13	6.42
6-Nitrobenzo[a]pyrene	(6.1)*	(4.8)

* Values in parantheses are not certified, but are given for information only.

SRM 1614 - Dioxin (2,3,7,8-TCDD in Isooctane)

Compound	Concentration	
	ng/g	ng/ml, 23° C
2,3,7,8-TCDD	98.3	67.8
2,3,7,8-TCDD-^{13}C	95.6	65.9

APPENDIX J: EXAMPLE OF A PROCEDURE FOR SPIKING WATER WITH CADMIUM

EXAMPLE OF A PROCEDURE FOR SPIKING WATER WITH CADMIUM

1. Obtain the standard, in this case cadmium, as either a primary metal or as an analytical reagent grade compound.

2. The final concentration of cadmium in the stock reagent solution will be 1,000 mg/ℓ or 0.1 percent in 0.01 N nitric acid (store stock solution in precleaned opaque liner polyethylene or Teflon at 4° C).

3. The following is a presentation of the calculations necessary to compute the amount of Cd $(NO_3)_2$ to be used:

	Molecular Weight
Cd	112.40
$(NO_3)_2$	124.01
Cd $(NO_3)_2$	236.41

The weight of Cd $(NO_3)_2$ required to yield 1.0 g Cd should now be computed:

$$\text{Weight of Cd } (NO_3) \text{ required} = \frac{(1.0 \text{ g Cd})(\text{Molecular weight of Cd } (NO_3)_2}{\text{Molecular weight of Cd}}$$

$$= \frac{1.0(236.41)}{112.40} = 2.1035$$

Enough Cd $(NO_3)_2$ should be oven-dried and desiccated to yield the 2.1035 g of Cd $(NO_3)_2$ that will yield 1.0 g Cd.

4. Dissolving the 2.1035 g of Cd $(NO_3)_2$ in 1,000 g of 0.01 N nitric acid equals 1,000 mg Cd/kg water or 1,000 mg/ℓ at 4° C.

5. One liter of representative water sample (measured volumetrically) should be taken for the standard addition procedure. The standard solution should be held to minimum volumes to avoid dilution effects (1 ml or less).

6. Final concentrations should be adjusted to give spike concentrations approximately twice that of the expected concentration.

7. The general equation for determining the concentration resulting from the spike is

Final concentration of spiked 1 ℓ of water

$$= \frac{(\mu g/ml \text{ of standard})(ml \text{ added})}{1 \ \ell}$$

$$= \mu g/\ell$$