



REGIONAL IMPLEMENTATION MANUAL

for the

EVALUATION OF DREDGED MATERIAL PROPOSED FOR DISPOSAL IN NEW ENGLAND WATERS

Prepared by

U.S. EPA NEW ENGLAND

and the

U.S. ARMY CORPS OF ENGINEERS, NEW ENGLAND DISTRICT

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PREFACE

This Regional Implementation Manual for the Evaluation of Dredged Material Proposed for Disposal in New England Waters, or RIM, was prepared by EPA Region 1, New England in cooperation with the New England District (CENAE) of the U.S. Army Corps of Engineers (Corps) to provide guidance for applicants proposing open-water disposal of dredged material in New England waters. Under the authorities of Section 103 of the Marine Protection, Research and Sanctuaries Act (for disposal seaward of the baseline of the territorial sea) and Section 404 of the Clean Water Act (for disposal inland and in near coastal waters landward of the baseline), the Corps and EPA have issued national guidance and testing requirements to evaluate dredged material for open water disposal. These national guidance manuals are called the "Green Book" ("Evaluation of Dredged Material Proposed for Ocean Disposal Testing Manual", EPA/USACE, 1991), and the "Inland Testing Manual" ("Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual", EPA/USACE, 1998). The RIM applies the national guidance to the New England area and supercedes the previous regional manual entitled "Guidance for Performing Tests on Dredged Material to be Disposed of in Open Waters" (EPA/NAE, 1989). The RIM provides New England-specific guidance on: permit application and coordination requirements; sampling methodologies; updated reference site locations; contaminants of concern and analytical reporting limits; and species and test conditions for biological testing.

This manual reflects advances in scientific methodologies and environmental evaluation since the publication of the 1989 manual. Specifically, this manual includes: additional contaminants of concern (e.g. organotins); PCB congener analysis; updated analytical methodologies and reporting limits (e.g. for PAHs, pesticides and dioxins); new test species and number of species tested for bioassays (e.g. the amphipod *Leptocheirus*); and a shift in duration of bioaccumulation tests from 10 to 28 days. One of the more important changes is that applicants must gain approval for Sampling and Analysis and Laboratory Quality Assurance plans that meet rigorous quality assurance and quality control requirements. Applicants also must submit sediment and tissue chemistry data electronically in specific data formats (available at <www.nae.usace.army.mil/reg/rim.htm>).

Consistent with our efforts to ensure coordination and consistency with other applicable federal and state laws and regulations, policies, requirements, and environmental practices, the RIM has also been reviewed by regional offices of the National Marine Fisheries Service and the U.S. Fish and Wildlife Service, and environmental resource agencies of the five coastal New England states. The draft RIM was publicly noticed in December 2002 and comments as appropriate were incorporated. This RIM is hereby approved by the following officials of the Corps and EPA and goes into effect on May 6, 2004:

Brian A. Green Lieutenant Colonel, Corps of Engineers Acting District Engineer Date

Robert W. Varney Regional Administrator EPA New England Date

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LIST OF ABBREVIATIONS AND ACRONYMS

| AED | Atlantic Ecology Division, EPA Research Lab, Narragansett, Rhode Island |
|-------|---|
| APHA | American Public Health Association |
| ASTM | American Society of Standards and Materials |
| CRM | Certified Reference Material |
| CWA | Clean Water Act |
| DM | Dredged Material |
| DOA | Department of the Army |
| ENG | U.S. Army Engineering Form |
| EPA | U.S. Environmental Protection Agency |
| FWS | U.S. Fish and Wildlife Service |
| GC/MS | Gas Chromatography/Mass Spectroscopy |
| HTL | High Tide Line |
| ITM | Inland Testing Manual |
| LC50 | Median Lethal Concentration |
| LCS | Laboratory Control Sample |
| LPC | Limiting Permissible Concentration |
| LIS | Long Island Sound |
| LQAP | Laboratory Quality Assurance Plan |
| MDL | Method Detection Limit |
| MHW | Mean High Water |
| MLLW | Mean Lower Low Water |
| MLW | Mean Low Water |
| MPRSA | Marine Protection, Research and Sanctuaries Act |
| CENAE | New England District, U.S. Army Corps of Engineers |
| NMFS | National Marine Fisheries Service |
| NOAA | National Oceanic and Atmospheric Administration |
| NYDEC | New York Department of Environmental Conservation |
| OHW | Ordinary High Water |
| PAH | Polycyclic Aromatic Hydrocarbon |
| PCB | Polychlorinated Biphenyl |
| PSEP | Puget Sound Estuary Program |
| ppb | parts per billion |
| ppm | parts per million |
| ppt | parts per thousand (used for salinity measurements) |
| pptr | parts per trillion |
| QA/QC | Quality Assurance/Quality Control |
| RIM | Regional Implementation Manual |
| RL | Reporting Limit |
| SAP | Sampling and Analysis Plan |
| SIM | Selected Ion Monitoring |
| SRM | Standard Reference Material |
| TBP | Theoretical Bioaccumulation Potential |
| TOC | Total Organic Carbon |
| USACE | U.S. Army Corps of Engineers |
| WQC | Water Quality Criteria |

1. INTRODUCTION

This Regional Implementation Manual (RIM) presents sediment testing guidelines and reporting requirements for applicants who wish to obtain a Department of Army permit from the New England District (CENAE) of the U.S. Army Corps of Engineers (Corps) for all projects (both private and federal navigation) involving the open water disposal of dredged material. This guidance is consistent with national guidance (described below) and has been approved by the U.S. Environmental Protection Agency (EPA) and the Corps in cooperation with the U.S. Fish and Wildlife Service (FWS), National Marine Fisheries Service (NMFS) and the various permitting and environmental resource agencies of the five coastal New England states: Maine, New Hampshire, Massachusetts, Rhode Island and Connecticut.

This manual implements the national testing guidelines under Section 103 of the Marine Protection, Research and Sanctuaries Act (MPRSA) (33 USC 1401 et seq.) and Section 404 of the Clean Water Act (CWA) (33 USC 1344 et seq.). The MPRSA governs (1) all disposal projects in New England ocean waters (seaward of the territorial sea baseline), and (2) disposal of dredged material in Long Island Sound of federal disposal projects of any amount or those non-federal disposal projects exceeding 25,000 cubic yards. In addition, Section 404 of the Clean Water Act regulates the disposal of dredged and fill materials into waters of the U.S. landward of the territorial sea baseline and fill material within the territorial sea. The guidance and requirements specified in this document will be used by the regulatory agencies for all disposal activities subject to Section 103 of the MPRSA (40 CFR Parts 227.6 and 227.13) and/or Section 404 of the Clean Water Act (40 CFR Parts 230.60 and 61).

The MPRSA requires that operations involving the transportation and discharge of dredged materials in ocean waters are to be evaluated to determine their potential impact to the marine environment. The proposed disposal must be evaluated through the use of criteria published by the EPA in Title 40 of the Code of Federal Regulations, Parts 220-228 (40 CFR 220-228). In accordance with Subsection 227.27 (b) of the regulations, EPA and the Corps developed a national testing manual to define procedures for evaluating the suitability of dredged material for ocean disposal that are based upon the testing requirements in the regulations. This national testing manual is entitled "Evaluation of Dredged Material Proposed for Ocean Disposal Testing Manual" and is commonly known as the "Green Book" (EPA/USACE, 1991). It replaced the first testing manual "Ecological Evaluation of Proposed Discharge of Dredged Material into Ocean Waters" (EPA/USACE, 1977).

Proposed disposal into waters covered under the CWA must be evaluated under the 404(b)(1) guidelines (40 CFR Parts 230.10 and 230.11). As specified in 40 CFR Parts 230.60 and 230.61, EPA and the Corps developed the national manual "Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Testing Manual" (commonly known as the "Inland Testing Manual", or ITM) (EPA/USACE, 1998).

The 1991 Green Book and 1998 ITM provided new and improved testing methods and contain revised guidance that reflects the regulatory experience gained since the 1977 testing manual was published. The Green Book, ITM and the companion Quality Assurance/Quality Control

(QA/QC) manual titled: "Guidance for Sampling and Analysis of Sediments, Water and Tissue for Dredged Material Evaluations: Chemical Evaluations" (EPA/USACE, 1995, commonly known as the "QA/QC manual") provide national guidance on the tiered-testing approach, sampling methodology, testing procedures, statistical methods and QA/QC.

This Regional Implementation Manual (RIM) applies the national guidance to the New England area, providing additional guidance agreed upon by EPA Region 1 and CENAE in cooperation with the above listed agencies. It replaces the previous regional manual entitled "Guidance for Performing Tests on Dredged Material to be Disposed of in Open Waters" (EPA/NAE, 1989) which implemented the 1977 national testing manual. This current manual provides needed supplementary guidance on: permit application requirements, data and reporting requirements, a list of the contaminants of concern, species for biological testing and specific procedural requirements agreed upon by state and federal agencies. This will avoid unnecessary confusion and possible delays or expenses through the submission of improper data. Except where noted, this document does not attempt to duplicate or replace the detailed information contained in the Green Book, ITM or the QA/QC manual. Instead it should be used in conjunction with these manuals to provide additional information or clarification when needed. Specific references to appropriate sections are provided.

New and more advanced testing procedures and guidelines are continually being developed and refined by the research and development laboratories of the EPA and the Corps. In addition, ongoing monitoring of designated disposal sites in New England waters under the CENAE Disposal Area Monitoring System (DAMOS) program can provide effects-based feedback to the regulatory agencies allowing them to make more refined, environmentally sensitive and efficient decisions regarding the acceptability of proposed open water disposal of dredged material. The DAMOS program can also determine whether any site-specific criteria are needed for a particular disposal site. As a result, this manual will be revised as needed to incorporate any necessary modifications of the testing guidance.

All application information, as discussed in the following sections, should be submitted by applicants to the CENAE office in Concord, Massachusetts. The CENAE will supply copies of the information to the other federal agencies including EPA, FWS and NMFS. Note that applicants are required to contact the appropriate state regulatory agency directly. The applicant should know that additional information may be required on a project by project basis.

Questions about this manual should be directed to:

U.S. Army Corps of Engineers New England District Regulatory Division 696 Virginia Road Concord, MA 01742-2751 (978) 318-8338

or

U.S. Environmental Protection Agency New England, Region 1 Office of Ecosystem Protection (CWQ) One Congress Street, Suite 1100 Boston, MA 02114-2023 (617) 918-1553

2. ADMINISTRATIVE REQUIREMENTS

2.1 General

When applying for a Department of the Army permit to dispose of dredged material into open water, the applicant will be required by CENAE to provide the information indicated below. This information represents the first of four information tiers used in the evaluation of dredged material. As discussed in Section 3, the evaluative processes in the remaining tiers (including biological testing of the materials proposed for dredging) may be necessary.

Additional guidance on preparing applications can be found in the most recent edition of CENAE's <u>GUIDE FOR PERMIT APPLICANTS</u>. Useful general application information, application forms, and sample project plans are available in the guide (also available on the CENAE website at <www.nae.usace.army.mil/>). For the most recent copy, contact the New England District Regulatory Division at 978-318-8338, or toll free at 800-343-4789 or 800-362-4367 if calling within Massachusetts.

Upon receipt of a permit application or pre-application inquiry, CENAE will assign a Regulatory Division Permits Project Manager who will serve as the applicant's point of contact throughout the review process. Information required for review by the CENAE and coordinating federal and state agencies include, but are not limited to, the following data:

- 1. A statement describing why the proposed dredging is required, if it is "new, or improvement" or "maintenance" dredging, and the area (square feet or acres) and volume (cubic yards) of material to be dredged. If the project is comprised of several "segments" (e.g., marina basin and an entrance channel), volume and area estimates should be provided separately for each. The side slope should be included in the area and volume estimates. The volume estimate(s) should be provided both with and without the dredged material associated with the maximum estimated overdredge. Current and proposed water depths should be described based on mean low water (MLW) or mean lower low water (MLLW).
- 2. Alternative disposal locations with information in sufficient detail to evaluate their potential for use. This should include a comprehensive survey of potential upland, beneficial use (e.g. beach nourishment) and open water sites. The names and addresses of nearby landfills or other available upland disposal sites should be provided. The availability of the immediate upland area should also be discussed. An explanation should also be provided on why no disposal options other than open water sites are practicable.
- 3. The date when the project was last dredged and any previous sediment and biological effects test data for this or nearby projects that would aid in typifying project sediments. In the absence of any previous test data, a description of the bottom material should be provided (e.g., rock, sand, vegetated, etc.).
- 4. Information on locations of outfalls, non-point sources of contaminants and any recent

contaminant spills must also be included as described in Chapters 2 and 8 of the ITM. These data may be obtained from sources such as state water pollution control agencies (e.g., Department of Environmental Protection), U.S. Coast Guard and harbormasters. The sources of all information must be properly documented.

- 5. Two legible copies of 8.5" X 11.0" drawings (Figure 1) including plan views and cross sections of the area to be dredged with the following information noted:
 - Proposed depth of dredging and proposed overdredge referenced to vertical datum (MLW or MLLW)
 - Dredge boundaries with area (sf) and volume (cy) for each dredge location, including side slope
 - Existing depths referenced to vertical datum (MLW or MLLW)
 - Shoreline/limits of waterways: high tide line (HTL), mean high water (MHW) and MLW
 - Ebb and flood in tidal waters and direction of flow in non-tidal waters
 - North arrow, numerical and graphic scale (avoid reduction and enlargement)
 - Outfall locations (e.g. industrial, stormwater and wastewater discharges)
 - Non-point sources of contaminants (parking lots, oil storage tanks, hazardous waste, etc.)
 - Proposed and historical sampling locations (if appropriate)
 - Separate vicinity/locus map with readily identifiable landmarks (USGS Quad Sheet photocopy or a local road map is acceptable)
 - Permit application plans should conform to other specifications as noted in the "GUIDE FOR PERMIT APPLICANTS."
- 6. Type of dredging equipment to be used (clamshell, hydraulic, etc.) and any unique handling procedures to be used, such as a sealed clamshell or special runback controls.
- 7. Proposed dredged material disposal site. The following information is required for each proposed disposal site, except for the authorized regional open water disposal sites:
 - Locus sheet
 - Detailed plan view
 - Information on any significant resources (e.g. shellfish beds, fish habitat, submerged aquatic vegetation, water supply wells) at and near the proposed site
 - Limits of regulated areas if applicable, e.g. waterbodies (ordinary high water (OHW), HTL and MHW) and wetlands
 - For open water sites, hydrological and physical information including currents, water depth, bottom sediment texture (grain size composition) and whether the site is a depositional or a dispersive site
- 8. Proposed beach nourishment site, if applicable. The following additional information is

required:

- HTL, MHW, MLW
- Delineation of any vegetated areas and/or resource areas in the vicinity (e.g. salt marsh, submerged aquative vegetation, wetlands, shellfish beds)
- Grain size composition of the beach sediment if available
- 9. Dewatering site. If the material is to be dewatered, the following information should be provided:
 - Description of the site
 - Locus sheet
 - Plan view and cross section of the dewatering site
 - Calculations used to determine the capacity of the dewatering area
 - Details of the methods to be used to control runback

2.2 Coordination

Early coordination with the CENAE Regulatory staff is required to determine the sediment contaminant analyses needed, and for development and approval of a project Sampling and Analysis Plan (SAP). The SAP includes proper sampling techniques, location and number of samples to be taken, associated quality assurance measures and other project-specific information on the actual field sampling effort (see Chapter 4). The applicant may request that CENAE develop a SAP, or the applicant may submit a SAP for CENAE approval.

Prior to any sampling and testing the applicant must also provide to CENAE a Laboratory Quality Assurance Plan (LQAP) for its data and analysis to be accepted for permit applications (unless previously submitted). The LQAP provides standard quality assurance/quality control (QA/QC) procedures used by the contractor laboratory. CENAE and EPA are currently programmatically reviewing LQAPs from laboratories performing testing for this regulatory program. A 24 month grace period will be allowed for existing labs to submit LQAPs from the effective date of this document. After that date, any new labs will be required to submit and have approved LQAPs before any project data will be accepted.

The federal permitting process (Figure 2) involves a comprehensive evaluation process and requires a multi-agency review of dredged material suitability determinations. Of prime importance is the interagency coordination process whereby CENAE coordinates fully with Federal agencies with resources of concern. EPA Region 1 has the authority to review, approve (for MPRSA projects only), or propose conditions upon permits for open water disposal, and NMFS reviews project evaluative steps and provides information on endangered species, Essential Fish Habitat and other biological resources. Applicants should also consult with the State Historic Preservation Office early in the permit process to ensure the proposed locations for dredging and disposal of dredged material are in compliance with Section 106 of the National Historic Preservation Act. Early coordination (at the application or pre-application stage) ensures that unnecessary delays do not become a factor in the review process.





FIGURE 2. Generalized coordination procedure for sediment suitability determination



3. TIERED TESTING

The tiered approach to testing consists of successive levels of investigation, each with increasing effort and complexity. This approach generates the information necessary to evaluate the proposed disposal of dredged material at an open water site. It provides for optimal use of resources by focusing the least effort on dredging operations where the potential (or lack thereof) for unacceptable adverse impact is clear, and expending the most effort on operations requiring more extensive investigation to determine the potential (or lack thereof) for impact. This approach is described in detail in Chapters 4 to 7 of the 1991 Green Book and Chapters 3 to 7 of the ITM. These chapters should be read thoroughly in either manual, depending upon the jurisdiction, to ensure a full understanding of all tiered testing requirements. A brief description of the tiered testing approach is presented below and illustrated in Figure 3. Prior to undertaking any testing, applicants must coordinate with their CENAE Project Manager.

3.1 Tier I - Review of Existing Information and Identification of Contaminants of Concern

The purpose of Tier I evaluations is to determine if existing information on the proposed dredged material is sufficient to demonstrate compliance with regulations and to determine contaminants of concern. A comprehensive review of existing and readily available information is required to make this determination. If existing test data are considered inadequate to evaluate the proposed project, new sediment chemical and/or biological testing are required.

3.2 Tier II - Water Column and Potential Bioaccumulation Analyses

Tier II consists of an evaluation of compliance with water quality criteria (WQC) using a numerical mixing model (Appendix B, Green Book; Appendix C, ITM) and an evaluation for potential bioaccumulation using calculations of Theoretical Bioaccumulation Potential (TBP; Section 5.2 of the ITM) for non-polar contaminants of concern. Sediment chemistry data are used for these analyses.

3.3 Tier III - Toxicity and Bioaccumulation Testing

Tier III testing is used to provide data for an impact assessment of the contaminants of concern through use of toxicity and bioaccumulation tests with appropriate, sensitive organisms (see Tables 6 and 7 for test organisms). Both water column toxicity testing and benthic toxicity testing are required. Bioaccumulation testing is used to determine the potential for uptake of sediment contaminants at the disposal site by benthic organisms.

3.4 Tier IV - Long Term Bioassays and Bioaccumulation Tests, Risk Evaluations and other case-specific testing/evaluations

Under unusual circumstances, such as when a unique resource or resource area is involved, it may be necessary to evaluate long-term effects of proposed dredged material on appropriate sensitive aquatic organisms, as well as human health risks. A risk assessment prepared by EPA Region 1 may be required to interpret bioaccumulation results. Because of the limited

availability of appropriate and widely accepted procedures, each test is selected to address specific concerns of each disposal operation (Section 7.1 Green Book; and Section 7 of the ITM). In a situation such as described, extremely close coordination with EPA and CENAE in all aspects of Tier IV testing is required.

FIGURE 3. Generalized tiered process for review of dredging projects



4. SAMPLING METHODOLOGY

The importance of a well-designed sampling program is underscored by the fact that an evaluation of the potential impacts of a proposed dredging project is only as complete and reliable as the sampling upon which it is based. The quality of information gathered through the tiered testing process is affected by the following sampling-related factors: a) collecting representative samples; b) using appropriate sampling techniques; and c) protecting or preserving the samples until they are tested. It is the responsibility of the applicant to ensure that samples taken for a proposed project meet the QA/QC requirements presented below and discussed in Chapter 8 of the Green Book and the ITM, and the QA/QC manual (EPA/USACE, 1995). *Failure to meet these requirements or follow any specified procedure without CENAE approval will likely cause rejection of the testing results. Applicants should always consult with CENAE and obtain approval before beginning any sampling effort.*

4.1 Development of a Project Sampling and Analysis Plan (SAP)

Applicants must have a project Sampling and Analysis Plan (SAP) (see Chapter 2) which together with the LQAP make up the Quality Assurance Project Plan. Applicants may submit a proposed sampling plan for approval to CENAE, or request a SAP be prepared by CENAE based on submitted information specified in Chapter 2. CENAE will develop and/or approve the SAP in coordination with the federal agencies (and state agencies if appropriate). CENAE will provide the approved SAP to the applicant, including the number and location of samples, the required analytes, reporting limits (RLs; see Chapter 5) and other project-specific information supplemental to the LQAP. The approved SAP must be implemented by the applicant. Any changes to the approved SAP must be approved in writing by CENAE prior to sampling.

Please note that applicants should not, under any circumstances, undertake field sampling and analysis without first coordinating with the CENAE and receiving an approved SAP from the CENAE.

If the applicant chooses to submit a SAP for CENAE approval, the following information must be included:

- a brief project description, contract lab name and address, and a letter stating when the LQAP was sent to CENAE;
- reference site and disposal site locations (see below);
- station-specific sampling procedures (including sampling gear and proposed positioning methodology) and description;
- sample handling/storage procedures; and
- analytical procedures and reporting limits (see Chapter 5).

4.2 Sampling of Proposed Dredged Material

Sediment samples must be collected according to the approved SAP. In the instances where vertical grain size homogeneity exists and the project depth is less than 2 feet, a grab sampler can be used if approved by the CENAE prior to field sampling. A core sampler should be employed in all other cases to ensure the samples are representative of materials to dredging depth, including expected overdepth. To ensure a sample is representative of a project, CENAE must approve the sampling apparatus. The type of equipment used to collect the samples should be noted as part of the project record. For example, if coring was used, the type of corer (gravity, vibracore, split spoon, borings, etc.) and the core liner (polycarbonate or butyrate, etc.) should be added to the field documentation. Core logs should be provided, with narratives describing relative grain sizes, color, odor, strata, core length and depth of penetration along with other pertinent sediment sampling observations.

In instances where significant distinct vertical stratification (at least 2.0 feet) is evident in samples, subsampling and testing of each layer (e.g. sand vs. silt) may be required to adequately characterize the materials. The cores must be inspected in the field for stratification. If the cores show significant stratification, subsamples must be made of each layer. CENAE is available for consultation on whether significant stratification is present. If there is neither time nor opportunity to contact the CENAE, then the applicant should take the subsamples, store them separately and bring them back to the laboratory. *The goal is to avoid compositing dissimilar sediment and to provide the best possible characterization of the material. This avoids misrepresenting the amount of contaminated material that may require special and, likely, more expensive disposal options.*

In situations where grain size analyses show samples to be comparable and samples represent a similar project segment(s), compositing of samples may be permitted. In all instances where compositing is contemplated, CENAE must review grain size data prior to any compositing, and will make the final decision on any compositing scheme. Should compositing be allowed, subsamples of the individual samples making up the composites must be archived by the testing laboratory until results of analyses have been reviewed by CENAE.

Care should be taken to avoid sample contamination from sampling gear, grease, ship winches or cables, airborne dust, vessel engine exhaust, cross contamination and improper subsampling procedures. Engines should be shut off during sampling, if possible. If not possible (due to boat traffic, type of workboat, currents, etc.), then the sampling effort should be performed upwind of the exhaust. It is recommended that core extrusion and sample mixing be performed in the laboratory. If on-board mixing is necessary, however, this effort must be performed away from exhaust fumes and any other sources of contamination. In addition, care must be taken to avoid cross contamination. All core samplers or other sampling devices must be appropriately cleaned between samples. The applicant must ensure that the workboat has room to store cores vertically out of the way from contamination and disturbance.

A sufficient sediment mass must be collected to meet the objectives of the sampling program. A minimum of approximately 1000 grams of sediment per sample must be collected for bulk

physical and chemical analyses. The mass of sediment will vary with grain size, density, core depth/diameter and should be assessed before sampling to ensure adequate mass. It should be noted that other types of analyses require greater masses; for example, bioaccumulation tests need a minimum of 7,500 grams per sample (see Chapter 7, Section 7.2). Sufficient material must be available for analyses and for partitioning of samples to meet archiving requirements cited in Table 5 of the QA/QC manual. The guidance specified in "Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses" (EPA, 2001d) should also be consulted.

The project samples must be taken at the precise locations required by the CENAE-approved SAP. Vessel positioning must be determined, using any of a number of techniques including GPS, Loran and surveying equipment. GPS systems need to be calibrated using known references. *In all cases, CENAE requires that sample location latitudes and longitudes be recorded and provided to CENAE for each sampling location for compatibility with the CENAE regional database*. All coordinate data should be reported in NAD 83 decimal minutes. Locational information for each sampling point should be recorded in the field on a Station Location Log, Sediment Sampling Log or similar document and be included as part of the QA/QC portion of the analytical results. Examples of these types of documents are included in Appendix A of the QA/QC manual.

4.3 Sediment Sample Handling, Preservation and Storage

The applicant is responsible for ensuring that the sampling, handling and preservation and storage procedures and the applicable QA/QC measures are followed for both sampling and analysis. These procedures must be adequately described in the approved SAP and the LQAP. The guidance specified in "Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses" (EPA, 2001d) should also be consulted.

Samples are subject to chemical, biological and physical changes as soon as they are collected. It is therefore imperative that, from initiation of collection activities until samples are analyzed, all applicable QA/QC procedures are followed.

Sample preservation should be accomplished onboard the collecting vessel whenever possible. If final preservation cannot occur onboard, an interim preservation technique that preserves the sample integrity should be employed. Onboard refrigeration can be accomplished with coolers and ice while samples that are to be frozen can be placed in coolers with dry ice. Sediment samples for grain size, elutriate and biological analysis should not be frozen but preserved at 4°C. Samples should not be allowed to dry. Additional information is given in Chapter 8 (Table 8-2) of the Green Book (EPA/USACE, 1991) or in the QA/QC manual (EPA/USACE, 1995).

In general, careful choice of sampling gear and containers should be made for each group of chemicals to be analyzed to avoid sample contamination. Prior to contact with samples, equipment and containers should be cleaned and rinsed. Specific methodologies and containers are discussed in EPA/USACE (1995) and EPA (2001d). Labels for the containers must be able to withstand environmental extremes and remain legible.

Sample containers should be filled to the top unless the sample is to be frozen, in which case room for expansion must be allowed. If subsamples are to be taken from the container, the container is best left about 3/4 full to allow for proper stirring.

Work should start as soon as possible on sediments so as not to exceed the holding time requirements (EPA/USACE, 1998). The time between sample collection and analysis should be minimized to maintain the integrity of the sample. The longer the sample is stored, the more difficult it becomes to accurately assess sample results; over time a sample may become increasingly toxic to bioassay organisms (due to ammonia or other constituents).

4.4 Sampling of Reference and Control Sediments

Sample handling, preservation and storage QA/QC requirements need to be followed (see EPA/USACE 1995 and EPA, 2001d) and are the same for reference and control sediments as those for the dredged material. **Reference samples, however, may be collected with grab samplers.**

Reference sampling sites are determined through an EPA Region 1 and CENAE cooperative program that designates reference locations for each active disposal site. The location of reference sampling sites for each established disposal site are shown below (in decimal minutes NAD 83). Current reference sampling sites will be indicated in the approved SAP. If these reference sampling sites are relocated, the updated coordinates will be included in the approved SAP. Testing laboratories are responsible for collection of control sediments for benthic effects evaluations.

| Rockland | 44° 07.1' N | 68° 58.70' W |
|---------------------------------|--------------|--------------|
| Portland | 43° 38.6' N | 69° 59.01' W |
| Cape Arundel | 43° 17.9' N | 70° 26.02' W |
| Massachusetts Bay | 42° 22.70' N | 70° 30.30' W |
| Cape Cod Bay | 41° 57.50' N | 70° 16.00' W |
| New London | 41° 16.7' N | 72° 02.0' W |
| Cornfield Shoals | 41° 15.63' N | 72° 13.32' W |
| Central Long Island Sound (LIS) | 41° 08.1' N | 72° 50.06' W |
| Western LIS | 40° 58.69' N | 73° 29.20' W |
| | | |

4.5 Sampling of Water

Sample handling, preservation and storage QA/QC requirements need to be followed (see EPA/USACE, 1995 and EPA, 2001d). Water samples must be collected with either a non-contaminating pump or a discrete water sampler following the guidance given in Sections 8.2.5 and 11.1.4 of both the Green Book and the ITM. Additional information regarding types of water to be collected and specific depths are as follows:

Dredging site water for preparation of the elutriate tests and water column toxicity tests shall be collected from the dredging site sediment sample locations avoiding any outfalls or other sources of pollution. For dredging sites <30 feet depth, one mid-depth sample shall be collected. For sites >30 feet depth, the sample should be a composite of near surface, mid-depth and near bottom samples (3 feet above the bottom).

Dilution water is used in water column toxicity tests to make up the required dilutions. It must be clean seawater, appropriately aged artificial seawater, or seawater collected from the disposal site reference location at near surface or mid-depth prior to collection of the sediment samples.

Control water is analogous to control sediment as it is used for water column toxicity control treatments. Control water must be the same water in which the test organisms are held prior to testing.

4.6 Sample Documentation

A complete field record of all procedures must be maintained including station locations, sample handling, preservation and storage procedures. Any circumstances potentially affecting the sampling must be noted as they may be necessary to explain a data anomaly.

The following information represents the minimum that must be placed on a sample label:

- Unique identifying code
- Location (station number) and depth
- Analysis or test to be performed
- Preservative and/or storage method
- Date and time of collection
- Special remarks if appropriate
- Initials or name of person doing the collecting

The information on the sample label represents the first step in a sample tracking (chain of custody) procedure. This procedure for tracking samples from collection through completion of analyses has to be in place prior to the initiation of sampling, with appropriate personnel assigned responsibility for the tracking and sample custody. Example sample labels and Chain of Custody forms are provided in Appendix A of EPA/USACE (1995).

As part of the chain-of-custody procedure and to insure an accurate evaluation of test results, sample designations used to identify sample locations in the field must be maintained throughout the process from sampling to data presentation. Records should include field log books, location of samples (latitude, longitude), positioning technology, sample labels, records of containers, time and conditions of storage. All sample containers and storage conditions must comply with the specifications in the Green Book, ITM, the QA/QC manual and EPA (2001d). Laboratories should keep records for a minimum of 5 years.

4.7 Data Reporting

All sampling data must be submitted to CENAE electronically and as hard copy. The required format for the electronic submission will be provided to the applicant when the SAP is approved, and is also available on the CENAE website (<www.nae.usace.army.mil/reg/rim.htm>). In addition, the applicant must provide completed Quality Control (QC) Summary Tables (Appendix II, also available on this website) and results of the QC analyses both as hard copy. This format is necessary to facilitate the project review process and to ensure completeness of the submittal. *Project data not submitted in the described formats will be considered incomplete and a resubmittal will be required*.

5. PHYSICAL AND CHEMICAL ANALYSIS OF SEDIMENTS

Testing is commonly required to characterize the physical and chemical properties of sediments proposed for dredging. The following information supplements Chapter 9 of the Green Book and the ITM as well as the QA/QC guidance manual.

Within 24 months of the effective date of this manual, each laboratory must have an approved Laboratory Quality Assurance Plan (LQAP) (see Chapter 2) on file with the CENAE. After that date, any new labs will be required to submit and have approved LQAPs before any project data will be accepted.

5.1 Initial Characterization of Sediment

As described in Chapter 4, all individual core samples must be visually inspected prior to their extrusion from the core liner in preparation for subsampling, homogenization or compositing. Each core must be described in terms of any discernible sediment strata characterized by changes in composition, texture, grain size, color and odor (e.g., sulfides, oil).

Sediment proposed for dredging and reference sediments must be analyzed for grain size distribution, total organic carbon (TOC) and total solids and percent moisture (Table 1). In addition, specific gravity, bulk density and Atterberg limits may be required on a case-by-case basis and are described in Section 5.3.

The grain-size analysis must be conducted according to the methods described in Plumb (1981) or ASTM (1998a) and reported as percentages retained by weight in the following size classes at a minimum:

- Gravel;
- Coarse Sand;
- Medium Sand;
- Fine Sand; and
- Silt/Clay (expressed as "Fines")

Gravel and sand fractions should be separated using the standard sieve sizes in Table 1 (ASTM 1998a, D 422-63). In addition to reporting the percentages of each size class, the applicant must graph the cumulative frequency percentages using the U.S. Army Engineering (ENG) Form 2087 or a similar form (Figure 4). There may be cases where silt and clay fractions will need to be distinguished. The CENAE will provide guidance, on a case-by-case basis, on whether it is needed. Both silt and clay fractions should be quantified by hydrometer (ASTM, 1998a), pipette or Coulter Counter (Plumb, 1981). Further analysis of other size classes may be required to evaluate suitability for beneficial use or other purposes.





Note that the results of the above physical analyses may be used to support compliance with one or more of the three exclusionary criteria in 40 CFR 227.13(b) for ocean disposal or support a determination that the material is not a carrier of contaminants under 40 CFR 230.60(a) for other open water disposal. If physical analyses show that the dredged material meets one or more of the exclusionary criteria, and if other pertinent, historical, and site-specific information can support the criteria, the material may be approved for disposal without further testing.

5.2 Chemical Analysis of Sediment

The chemicals of concern routinely required are listed in Tables 2 and 3. Table I-1 in Appendix I lists additional project-specific contaminants of concern. The routine metals, PAHs, PCBs, and pesticides listed in Tables 2 and 3 were chosen based on their toxicity, their persistence in the environment, their ability to bioaccumulate and their widespread and consistent occurrence in New England estuarine, marine and freshwater sediments and organisms.

The Reporting Limits (RLs) listed in Tables 2 and 3 have been set between the lowest technically feasible quantitation level for routine analytical methods (Method Detection Limit) and available background concentrations at reference areas in the vicinity of the disposal sites. As a routine data acceptance criterion the Method Detection Limits (MDLs) for each analyte should be three to five times below the listed RLs (see below). MDLs are calculated using the method described below and must be performed at a minimum every 12 months. Achieving these Reporting Limits and Method Detection Limits is critical to providing a consistent and accurate quantitation of contaminants of concern and provides confidence in measured values at concentrations typical of areas near but unaffected by the disposal site or other pollution sources.

Method Detection Limit (MDL) is defined as:

A statistical determination based on measured variance that defines the minimum concentration of a substance that can be detected with 99% confidence that the analyte concentration is greater than zero. In other words, that the analyte can be qualitatively detected above signal noise. *Any analytes not detected (below the MDL) should be reported as the MDL and qualified with a "U*". Detection limits are analyte- and matrix-specific and may also be instrument- and laboratory-dependent (see below).

The procedure described below, based on 40 CFR Part 136, Appendix B, must be followed to verify the MDL for samples collected for each approved Sampling and Analysis Plan. This MDL verification must be submitted with the data or performed on a similar matrix within the previous year.

Select one representative relatively uncontaminated sample for each matrix and spike it with the analytes of concern so that the resulting concentration is between 1 and 5 times the RLs listed in Tables 2, 3, 5, 8 and Table I-1 (Appendix I). Prepare and analyze a total of seven spiked replicates of the chosen representative sample. Calculate the sample standard deviation (in concentration units) of the seven measurements for each analyte of concern. This value must then be multiplied by 3.143 and reported as the MDL.

Reporting Limit (RL) is defined as:

The minimum concentration of an analyte or category of analytes in a specific matrix (e.g. sediment) that can be identified and quantified above the MDL (usually three to five times) and within specified limits of precision and bias during routine analytical operating conditions, adjusted for sample processing volumes and factors (such as dilution). The reporting limit is based on the lowest standard in the calibration curve. *Quantitative measurement at the MDL is inaccurate and therefore data reported less than the Reporting Limit (RL, see below) and greater than or equal to the MDL should be qualified with a "J" as estimated.*

As noted in Tables 2 and 3, the specified methods and the EPA guidelines on clean metals techniques (EPA, 1996a,b,c,d) are not required, as other acceptable methods are available. Whichever method is used, it is the applicant's responsibility to meet the reporting limits and the specified performance standards in the attached QC Summary Tables (Tables II-1 through II-7, Appendix II). These performance standards assess accuracy, as measured by Standard Reference Material (SRM), and precision, as measured by duplicates and matrix spike duplicates, for the contaminant groups listed in Tables 2 and 3 and Appendix I. Each applicant must demonstrate that any new lab they choose can meet these specifications prior to the analysis of any samples by the approval of an LQAP (see Chapter 4). Some labs have had difficulties in the past meeting the required reporting limits because of inappropriate sample preparation and clean-up procedures to remove interfering substances typically found in marine sediments (e.g., sulfides). Appropriate sample preparation, clean-up and analytical methods have been developed for estuarine/marine sediments by NOAA (1993) and the EPA research laboratory at Narragansett, RI (EPA, 1993). These are available from EPA Region 1 upon request. If the Reporting Limits cannot be attained, a detailed explanation must accompany the data providing the reasons for not attaining the required reporting limits. Re-analysis may be necessary.

The concentration, reporting limit and method detection limit for each of the following analytes on a dry weight basis should be reported as: ppm for metals, ppb for organics, parts per trillion (pptr) for dioxins/furans and dioxin-like PCBs. Total organic carbon (TOC) and percent moisture, used to calculate dry weight concentrations, must also be reported. The format for reporting is discussed in Sections 4.6 and 5.5.

As discussed in Section 9.3.2 of the Green Book, capillary gas chromatography with electron capture detection is recommended for analysis of PCBs and pesticides, whereas GC/MS in the Selected Ion Monitoring (SIM) mode is recommended for the PAHs and other semi-volatiles to meet the RLs. Second column confirmation of pesticides is required. Such confirmation for PCBs is recommended but not required at this time. The eighteen PCB congeners (listed in Table 3) are those analyzed in the NOAA National Status and Trends Program (NOAA, 1991). Additional congeners such as the non-ortho, mono-ortho and di-ortho dioxin-like PCBs (e.g., PCB congeners 77, 126, 169) may be required when dioxin is a contaminant of concern.

Total organic carbon (Table 3) must be analyzed on all samples and subsamples in duplicate in addition to a SRM or laboratory control sample (LCS).

The CENAE may require analysis of additional contaminants of concern other than those listed in Tables 2 and 3 if they are identified in the Tier I review. These remaining pollutants and other potential contaminants of concern and acceptable RLs are listed in Appendix I. Required analyses will be documented in the approved SAP.

As a general rule, Gas Chromatography/Mass Spectroscopy (GC/MS) chromatograms must be scrutinized for unexpected or unusual spikes of compounds not included on the target analyte list. These compounds should be tentatively identified and reported. The intent is to provide a screen for any potentially ecologically adverse contaminants that were unanticipated when the target analyte list was developed.

5.3 Additional Physical Characterization of Sediment

Additional characterization of the sediments may be required on a case-by-case basis for modeling and geotechnical evaluations. These include specific gravity, bulk density and Atterberg Limits (Table 4). Specific gravity should be measured following APHA (1995), ASTM (1998b) or Plumb (1981). Bulk density of sediment should be determined according to Klute (1986) or DOA (1980). Atterberg Limits may be required to assess the relative cohesiveness of the sediment. The procedures are outlined in ASTM (1998c). The plastic/liquid limits and plasticity index must be reported on ENG Forms 3838 and 4334 (Appendix III), respectively, or a facsimile.

5.4 Quality Control Measures

The applicant must submit documentation of all QC measures performed during analysis of the samples using the QC Summary Tables in Appendix II. *If any of the control limit criteria are exceeded, the sampling results may not be accepted.* All QA/QC for Dioxin/Furan analyses (listed in Appendix I-1) must be documented according to the methods described in EPA Method 1613. The following analytical QC measures must be performed for the above referenced methods.

(a) *Physical Analyses:* The following QC checks are required for physical analyses (grain size, TOC and percent moisture) of sediments, as appropriate:

- Sample duplicate
- Analysis of SRM (for TOC only)

(b) Chemical Analyses: The following QC checks are required for chemical analyses of sediments:

- Initial calibration
- Calculation of MDLs

- Blind analysis of spiked or performance evaluation material for calibration verification
- Continuing calibration checks
- Analysis of SRMs or LCSs
- Method Blank
- Matrix Spike
- Matrix Spike Duplicate
- Analytical replicates
- Surrogates
- Internal standards

(c) Detection and Reporting Limits: The Method Detection and Reporting limits used in this manual are defined in Section 5.2, and the Reporting Limits for sediment chemistry are listed in Tables 1 to 4.

5.5 Data Reporting

All physical and chemical sediment data must be submitted to CENAE electronically and as hard copy. The required format for the electronic submission will be provided to the applicant when the SAP is approved, and is also available on the CENAE website

(<www.nae.usace.army.mil/reg/rim.htm>). In addition, the applicant must provide completed Quality Control (QC) Summary Tables (Appendix II, also available on this website) and results of the QC analyses both as hard copy. This format is necessary to facilitate the project review process and to ensure completeness of the submittal. *Project data not submitted in the described formats will be considered incomplete and a resubmittal will be required.*

The applicants may submit their own data summaries and analyses; however, they must also submit the original data and copies of sampling logs so that the CENAE and EPA can conduct independent analyses. All submitted data must be clearly presented and traceable to the original samples and subsamples. *Suitability determinations will not be issued based on an applicant's data analysis alone.*

TABLE 1. Parameters used for the physical characterization of sediments

| <u>Parameter</u> | Method | Measure/Quantitation limit | | |
|-----------------------------|--|--|--|--|
| Grain Size Distribution | Plumb, 1981; ASTM, 1998a | | | |
| Gravel (> 4.75mm) | | Retained on No. 4 Sieve | | |
| Coarse Sand (2.0 - 4.75) | nm) | Passing through No. 4 and retained on No. 10 Sieve | | |
| Medium Sand (0.425 - 2 | .0mm) | Passing through No. 10 and retained on No. 40 Sieve | | |
| Fine Sand (0.075 - 0.425mm) | | Passing through No. 40 and retained on No. 200 Sieve | | |
| Silt (0.005 - 0.075mm) | | As determined by Hydrometer, Pipette or Coulter Counter | | |
| Clay (< 0.005mm) | | As determined by Hydrometer, Pipette or Coulter Counter | | |
| Percent Moisture | Plumb, 1981; APHA, 1995 | 1.0% | | |
| Total Organic Carbon | Plumb, 1981; EPA, 1992; PSEP, 1986 | 0.1 % | | |

| <u>Metal</u> | Analytical <u>Method(s)¹</u> | Reporting <u>Limit (ppm)</u> |
|--------------|--|---------------------------------|
| Arsenic | 6010B, 6020, 7060, 7061 | 0.4 |
| Cadmium | 6010B, 6020, 7130, 7131 | 0.07 |
| Chromium | 6010B, 6020, 7190, 7191 | 0.5 |
| Copper | 6010B, 6020, 7210 | 0.5 |
| Lead | 6010B, 6020, 7420, 7421 | 0.5 |
| Mercury | 7471 | 0.02 |
| Nickel | 6010B, 6020, 7520 | 0.5 |
| Zinc | 6010B, 6020, 7950 | 1.0 |

 TABLE 2. Metal contaminants of concern, recommended analytical methods and reporting limits (dry weight) routinely analyzed in sediments

1 The specified methods are recommendations only, based on the Green Book, ITM and the QA/QC manual (EPA/USACE, 1995). Other acceptable methodologies capable of meeting the RLs can be used. Sample preparation methodology (e.g. extraction and cleanup) and sample size may need to be modified to achieve the required reporting limits.

 TABLE 3. Organic contaminants of concern, recommended analytical methods and reporting limits (dry weight) routinely analyzed in sediments

| <u>Contaminant</u> | Analytical <u>Method(s)¹</u> | Reporting <u>Limit (ppb)</u> |
|--|--|---------------------------------|
| PAHs | 8270C-SIM | 10 ppb ² |
| Acenaphthene Acenaphthylene Anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenzo(a,h)anthracene Fluoranthene Fluorene Indeno(1,2,3-c,d)pyrene Naphthalene Phenanthrene Pyrene | | |
| Pesticides | NOAA, 1993; 8081B | 1 ppb ² |
| Aldrin cis- and trans-Chlordane cis- and trans-Nonachlor Oxychlordane 4,4'-DDT, DDE, DDD Dieldrin alpha- and beta-Endosulfan Endrin Heptachlor Heptachlor Heptachlor epoxide Hexachlorobenzene Lindane Methoxychlor | | |
| Toxaphene | | 25 ppb |

 TABLE 3 (continued). Organic contaminants of concern, recommended analytical methods and reporting limits (dry weight) routinely analyzed in sediments

| <u>Contaminant</u> | Analytical <u>Method(s)</u> | Reporting <u>Limit (ppb)</u> |
|-----------------------------------|---------------------------------|---------------------------------|
| PCB Congeners ³ | NOAA, 1993; 8082A | 1 ppb ² |
| 8* | 2,4' diCB | |
| 18* | 2,2',5 triCB | |
| 28* | 2,4,4' triCB | |
| 44* | 2,2',3,5' tetraCB | |
| 49 | 2,2',4',5 tetraCB | |
| 52* | 2,2',5,5' tetraCB | |
| 66* | 2,3',4,4' tetraCB | |
| 87 | 2,2',3,4,5' pentaCB | |
| 101* | 2,2',4,5,5' pentaCB | |
| 105* | 2,3,3',4,4' pentaCB | |
| 118* | 2,3',4,4',5 pentaCB | |
| 128* | 2,2',3,3',4,4' hexaCB | |
| 138* | 2,2',3,4,4',5' hexaCB | |
| 153* | 2,2',4,4',5,5' hexaCB | |
| 170* | 2,2',3,3',4,4',5 heptaCB | |
| 180* | 2,2',3,4,4',5,5' heptaCB | |
| 183 | 2,2',3,4,4',5'6 heptaCB | |
| 184 | 2,2',3,4,4',6,6' heptaCB | |
| 187* | 2,2',3,4',5,5',6 heptaCB | |
| 195* | 2,2',3,3',4,4',5,6 octaCB | |
| 206* | 2,2',3,3',4,4',5,5',6 nonaCB | |
| 209* | 2,2',3,3',4,4',5,5',6,6' decaCB | |

- 1 The specified methods are recommendations only, based on the Green Book, ITM and the QA/QC manual (EPA/USACE, 1995). Other acceptable methodologies capable of meeting the RLs can be used. Sample preparation methodology (i.e., extraction and cleanup) (EPA 1993; NOAA 1993) and sample size may need to be modified to achieve the required reporting limits.
- 2 Applies to each analyte listed below unless otherwise noted.
- For numerical mixing evaluations, total PCBs are to be estimated based on the following: Total = 2 X [sum of 18 NOAA summation congeners indicated with a *] (T. Wade, personal communication). For values below the MDL, use one half the MDL; for values between the MDL and the RL use estimated values.

TABLE 4. Additional parameters used for the physical characterization of sediments

| <u>Parameter</u> | Analytical <u>Method(s)</u> | <u>Reporting Limit</u> |
|---|--|------------------------|
| Specific Gravity | Plumb, 1981 ASTM, 1998b APHA, 1995 | 0.01 |
| Bulk Density | Klute, 1986 DOA, 1980 | 0.01 g/cm ³ |
| Atterberg Limits | ASTM, 1998c | |
| Liquid Limit Plastic Limit Plasticity Index | | |

6. WATER COLUMN EVALUATION

6.1 Tier II - Compliance with Water Quality Criteria/Standards

The discharge of dredged material into the water column and resuspension at an open water disposal site may introduce sediment contaminants into the water column. As required in 40 CFR 227.6 (c)(1) and 40 CFR 230.10(b)(1), the discharge must be in compliance with marine water quality criteria after allowance for mixing for discharges in federal waters and state water quality standards for discharges in state waters, if applicable. Based on 40 CFR 227.6, compliance with marine aquatic life water quality criteria or state water quality standards must be evaluated for every discharge in federal or state waters. The federal criteria are shown in Table 5. State water quality requirements for dredged material discharges vary with each state. Each appropriate state environmental regulatory agency, in coordination with CENAE, will assess compliance with applicable state standards using the data described below. General guidance on water column evaluations is provided in the Green Book (Sections 9.4 and 10.1) and the QA/QC manual. Evaluation is a two step process.

Step 1: Evaluation for compliance with Water Quality Criteria

As a first step in evaluating compliance, CENAE uses the dry weight sediment concentrations of listed contaminants which assumes a total release from the sediments to the water column as described in Section 10.1.1 of the Green Book and Section 5.1 of the ITM. The model used is described below in Section 6.4. As discussed in those sections, the analysis need only be run for the contaminant of concern that requires the greatest dilution for compliance. If the modeled discharge meets the water quality criteria (WQC; Table 5), then no further analysis is needed. If the analysis shows that the discharge exceeds the criteria, then the standard elutriate test, as described in Step 2, must be performed. Disposal site water values are used in the calculation to determine WQC compliance, or, existing data (provided by CENAE) in the vicinity of the disposal site may be substituted.

Step 2: Standard Elutriate Analysis

The dredged-material elutriate preparation is conducted according to the methods presented in Section 10.1.2.1 of the ITM ("Standard Elutriate Preparation"). The elutriate is prepared with approximately one liter of homogenized dredged material mixed with overlying water from the dredged material site in a 1 to 4 volumetric ratio. To evaluate water quality criteria in the liquid phase, the elutriate water must be centrifuged to remove particulates. The chemical analysis of the elutriate and disposal site water is discussed in Section 9.4 of the ITM ("Chemical Analysis of Water"). Disposal site water values are used in the calculation to determine WQC compliance, or existing data (provided by the CENAE) in the vicinity of the disposal site may be substituted.

At a minimum, chemical analysis must be conducted for the inorganic and organic analytes given in Table 5. Additional contaminants of concern may be requested for specific projects.

Table 5 provides the recommended methods and required Reporting Limits (RLs) for each contaminant of concern. So-called "clean" techniques for sampling (EPA, 1995a) and analyses of metals are currently available from EPA and are listed in Table 5. For extraction and analysis of PCB congeners, the NYDEC method (NYDEC, 1991) is also recommended. The eighteen PCB congeners are listed in Table 3. If there is doubt about meeting the RLs, the applicant should contact CENAE before any analyses are performed.

Particular note should be taken of the volume of the water samples required to meet the RLs. As a general rule, a minimum of one liter of elutriate should be prepared for metals analysis. One liter of elutriate should be analyzed for organic compounds. Larger samples are recommended since there should be enough left over in case repeat analysis is required and for QC checks. Additional clean-up steps may be necessary for the organics. If the applicant collects disposal site water for the mixing evaluation (see Section 6.4 below), sufficient volume of water is needed to meet the Reporting Limits in Table 5, especially for the organics. An example procedure for collecting large field samples can be found in Appendix IV.

6.2 Tier III - Water Column Evaluations

Tier III water column tests evaluate the potential for toxicity of the dissolved and suspended portions of the dredged material that remain in the water column after discharge of the dredged material. The water column bioassays are run if the Tier II evaluations are inconclusive: i.e., there are no WQC for all contaminants of concern or there is reason to suspect additive or synergistic effects among the contaminants. The Tier III water column tests involve exposing fish, pelagic crustaceans and planktonic invertebrate larvae to a dilution series containing dissolved and suspended components of the proposed dredged material. Disposal site water, or clean or artificially aged seawater (see below) is used as the dilution water for the tests. An overview of the Tier III water column evaluations is presented in the Green Book and the ITM under Section 6.1 in both documents.

Technical guidance for performing the tests is provided in Section 11.1 of the ITM (Tier III: Water Column Toxicity Tests). CENAE will specify to the applicant which species in Table 6 of this manual will be required for these tests. Three series of tests are necessary; tests must be run using a fish (Menidia sp., Cyprinodon variegatus), a crustacean (the mysid shrimp, Americanysis bahia) and a planktonic larvae (bivalve or echinoderm). The mysids should be fed as prescribed by EPA (1991b) or ASTM (1998d,e). Bivalve larvae and silversides must not be fed (ASTM 1998d,e,f). Test duration is generally 96 hours except planktonic larvae which is typically 48 hours. Samples for the standard elutriate test and the toxicity test can be prepared from the same sediment-water mixture. The procedure for preparing the water column toxicity test sample is given in Section 11.1.4 of the ITM with the following modifications (italicized). In cases where the salinity of the disposal site water is detrimental to the health of the test organism (too low), all the toxicity water samples must be prepared using clean seawater. The necessary dilutions may be made using water collected from clean seawater or aged artificial seawater. Each series should include 100%, 50%, and 10% treatments and a 0% treatment (=100% dilution-water treatment). Clean seawater in which the organisms were held prior to testing must be run as a control. If the diluent is the same water the organisms are held in prior to testing, then the control and 0% treatment are one and the same. Some fine-grained sediments can create turbidity in the test water even after settling. In this case, the ITM Section 11.1.4 allows mild centrifugation "...until the suspension is clear enough at the first observation time for the organisms to be visible in the testing chamber."

For fish and mysid shrimp bioassays, a minimum of five replicates per treatment concentration and a minimum of 10 organisms per replicate are required. The applicant should ensure that organisms are not overcrowded in the test chambers which can stress the organisms and falsely influence the results. The number of surviving fish and mysid shrimp for each replicate must be recorded at 0, 1 to 2 hours, 24, 48, 72 and 96 hours. Dead or unresponsive organisms may be removed and replaced at the first observations, but not at any subsequent observations. Dead organisms should be counted and removed daily. Observations of organism behavior and activity must be recorded daily during the test.

For the larvae bioassays, a minimum of five replicates per treatment is also required. A suspension of fertilized eggs is used in the preparation of the test solutions. The suspensions containing bivalve larvae should contain 20 to 30 embryos/mL whereas the suspensions containing sea urchin larvae should contain 2000 embryos/mL. For the bivalve water column toxicity test, the ASTM (1998f) protocol should be followed. For the sea urchin larvae test, the procedures in Appendix V (EPA/AED, 1996) should be followed. A light box or dissecting microscope may be used to record the number of live animals; use of an image analyzer as discussed in this procedure is not required here. For the larval test, centrifugation of a turbid supernatant is not necessary and should not be performed. The test is terminated in 48 to 72 hours. At this time, the larvae in the 0% treatment should have reached the appropriate stage of development (straight hinge – D shape for bivalves and plutei for the sea urchin).

For all test organisms, any sublethal effects such as physical or behavioral anomalies must also be reported. Daily water quality records must be kept for salinity, temperature, dissolved oxygen (DO) and pH for each test dilution.

6.3 Quality Control Measures

The applicant must submit documentation of all quality control measures performed during analysis of the samples using the QC Summary Tables in Appendix II. If any of the control limit criteria are exceeded, the data may not be accepted. The following analytical QC measures must be performed for the above referenced methods:

(a) Water Chemistry: The following QC checks are required for chemical analyses of water:

- Initial calibration
- Calculation of MDLs
- Blind analysis of spiked or performance evaluation material for calibration verification

- Continuing calibration checks
- Analysis of SRMs or LCSs
- Method Blank
- Matrix Spike
- Matrix Spike Duplicate
- Analytical replicates
- Surrogates
- Internal standards

Both elutriate (made up of dredging site water and sediments to be dredged) and disposal site water (if collected) should be tested in triplicate.

(b) Water Column Toxicity Tests: All bioassays must be performed under the conditions specified in each of the test species sheets in Appendix VI in either natural seawater or a synthetic seawater adjusted to salinity appropriate for the test species and disposal site (generally 25 to 30 ppt).

The survival rate requirements in the Control treatments must be achieved. Failure to meet the applicable requirements below will likely invalidate the testing procedures and require retesting of the control and test samples.

Control mortality requirements: ≤10% mean of replicates

Control abnormality requirements: ≤30% for oyster and mussel larvae ≤40% for clam larvae ≤30% for sea urchin larvae

(c) Detection and Reporting Limits: The Method Detection and Reporting limits used in this manual are defined in Section 5.2, and the Reporting Limits for water chemistry are listed in Table 5.

6.4 Data reporting

All chemical water chemistry and toxicity data must be submitted to CENAE electronically and as hard copy. The required format for the electronic submission is available on the CENAE website (<www.nae.usace.army.mil/reg/rim.htm>). In addition, the applicant must provide completed Quality Control (QC) Summary Tables (Appendix II, also available on this website) and results of the QC analyses both as hard copy. This format is necessary to facilitate the project review process and to ensure completeness of the submittal. *Project data not submitted in the described formats will be considered incomplete and a resubmittal will be required*.

The applicants may submit their own data summaries and analyses; however, they must also submit the original data and copies of sampling logs so that the CENAE and EPA can conduct

independent analyses. All submitted data must be clearly presented and traceable to the original samples and subsamples. *Suitability determinations will not be issued based on an applicant's data analysis alone.*

6.5 Numerical Models for Initial-mixing Evaluations

This section describes how CENAE uses numerical models to evaluate testing results from water column bioassays. Initial-mixing evaluations for compliance with water quality criteria and toxicity will be performed by CENAE as part of their assessment of each project; *applicants or their agents do not need to run the models*. The following information supplements the national guidance in the Green Book Appendix B and in the ITM Appendix C: Evaluation of Mixing.

Numerical models are components of the Tiers II and III water column evaluations. The model used, STFATE, is contained in the Automated Dredging and Disposal Alternatives Management System (ADDAMS) from the ITM (not referenced in the 1991 Green Book). The updated model is available for unrestricted distribution from the U.S. Army Corps of Engineers Waterways Experiment Station Environmental Laboratory website

(<http://www.wes.army.mil/el/elmodels/index.html>) and can be run on IBM®-compatible personal computers.

STFATE is run only for the contaminant of concern that requires the greatest dilution. If the contaminant requiring the greatest dilution is shown to meet the Limiting Permissible Concentration (LPC), all of the other contaminants that require less dilution will also meet the LPC.

STFATE computes the movement of dredged material from an instantaneous dump and from a hopper dredge that falls as a hemispherical cloud. To properly apply this model, the total time required for the dredged material to leave the disposal vessel should not be greater than the time required for the material to reach the bottom. The model applies to both split-hull barge and hopper disposal.

This model accounts for the physical processes that determine the short-term fate of dredged material in the water column as it is disposed at open-water sites. The model assumes that the dredged material behaves as a dense liquid, and simulates the movement of the disposed material as it falls through the water column and spreads over the bottom. It does not account for resuspension or other long-term post-disposal phenomena on the water column or benthic environment.

Input data for the model are grouped into the following general areas:

- Description of the disposal operation
- Description of the disposal site
- Description of the dredged materials
- Model coefficients
- Controls for input, execution, and output

Table C-2 in the ITM (Appendix C: Evaluation of Mixing) lists the necessary input parameters and their corresponding units. Applicants must provide the following parameters: volume of dredged material in barge, vessel course and speed, barge length and width, and post-disposal draft of barge. Additional descriptions and guidance for selection of values for many of the model parameters are provided in Appendix C and directly on-line in ADDAMS.

For discharge in federal waters, the results of the toxicity test will be used to determine compliance with the LPC. The results of the water column tests are used to calculate the median lethal concentration (LC50). The LPC for the dredged material is 1% of the LC50. If the numerical mixing model predicts that the concentration of dredged material in the water column will not exceed 1% of the LC50 concentration either outside the disposal site or within the disposal site 4 hours after discharge of the dredged material, the proposed discharge of dredged material meets the water column LPC. If either of these criteria are not met, the dredged material does not meet the water column LPC. For compliance of discharges in state waters, general guidelines are explained in Section 11.1.6 and Appendix C of the ITM. The state environmental regulatory agency needs to be consulted to determine the mixing requirements for compliance with the water quality criteria in that state. Such mixing guidelines can vary with each state.

 TABLE 5. Required contaminants, recommended analytical methods, reporting limits and federal water quality criteria used in water quality criteria compliance determination

| <u>Contai</u> | <u>minant</u> | Analytical <u>Method(s)</u> | Reporting <u>Limit(ug/l)</u> | Federal Water <u>Quality Criterion (ug/l)</u> |
|---------------|-------------------------|--------------------------------|---------------------------------|---|
| Metals | 5 ^{1,2} | | | |
| | Arsenic | 200.9, 1632 | 1 | 69 |
| | Cadmium | 200.9, 1637 | 1 | 42 |
| | Chromium(VI) | 218.6, 1636 | 1 | 1100 |
| | Copper | 200.9, 1639, 1640 | 0.6 | 4.8 |
| | Lead | 200.9, 1639, 1640 | 1 | 210 |
| | Mercury | $245.7, 1631^3$ | 0.4 | 1.8 |
| | Nickel | 200.9, 1639, 1640 | 1 | 74 |
| | Selenium | 200.9, 1639 | 1 | 290 |
| | Silver | 200.9 | 0.5 | 1.9 |
| | Zinc | 200.9, 1639 | 1 | 90 |

1 Determined as "total recoverable metals."

- 2 Except for chromium and mercury, samples can be digested by Method 200.2 (EPA, 1991) and extracted by chelation/extraction such as described under "Metals-14" S 9.2 (EPA, 1979, revised 1983), prior to analysis by Method 200.9. EPA Clean metal techniques (1600 series) are described in EPA (1995a,b,c) and EPA (1996a,b,c,d).
- 3 Bloom and Crecelius (1983) method for determining mercury concentrations.

TABLE 5 (continued). Required contaminants, recommended analytical methods, reporting limits and federal water quality criteria used in water quality criteria compliance determination

| <u>Contaminant</u> Pesticides | | Analytical <u>Method(s)</u> | Reporting <u>Limit(ug/l)</u> | Federal Water <u>Quality Criterion (ug/l)</u> |
|--|-------------------------|--|--|--|
| | | 3510B, 8081B ⁴ | | |
| Aldrin Chlordane Chloropyrifo Dieldrin 4,4'-DDT alpha- and bo Endrin Heptachlor Heptachlor e Lindane Toxaphene | s eta-Endo poxide | osulfan | $\begin{array}{c} 0.26 \\ 0.02 \\ 0.002 \\ 0.14 \\ 0.03 \\ 0.007 \\ 0.007 \\ 0.01 \\ 0.01 \\ 0.26 \\ 0.04 \end{array}$ | $\begin{array}{c} 1.3\\ 0.09\\ 0.011\\ 0.71\\ 0.13\\ 0.034\\ 0.037\\ 0.053\\ 1.3\\ 0.21 \end{array}$ |
| Industrial Chemica | ls | | | |
| PCBs ⁴ Pentachlorop | ohenol | 3510B ⁴ , 8082A 3510B, 8270C | 0.006 2.60 | 0.03 13 |

4 Pesticides and PCBs can be extracted from the water by Methods 3510B and analyzed by Method 8081A (EPA, 1986); PCB congener analysis by NYDEC (1991) is also recommended.

| <u>Organism(s)</u> | <u>Scientific Name</u> | Typical Test Duration |
|--|---|---|
| Fish: | | 96 hours |
| Silverside Sheepshead minnow | Menidia.menidia or M. beryl Cyprinodon variegatus | llina |
| Mysid shrimp | Americamysis bahia | 96 hours |
| Planktonic larvae: | | 48 to 72 hours |
| Blue mussel American oyster Hard clam Coot clam Sea urchin | Mytilus edulis Crassostrea virginica Mercenaria mercenaria Mulinia lateralis Arbacia punctulata | |
| | Organism(s)Fish:Silverside Sheepshead minnowMysid shrimpPlanktonic larvae:Blue mussel American oyster Hard clam Coot clam Sea urchin | Organism(s)Scientific NameFish:Silverside Sheepshead minnowMenidia.menidia or M. beryh Cyprinodon variegatusMysid shrimpAmericamysis bahiaPlanktonic larvae:Blue mussel American oysterMytilus edulis Crassostrea virginica Mercenaria mercenaria Mulinia lateralis Anbacia punctulata |

TABLE 6. Organisms required for the water column bioassay¹

1 One type of organism must be tested from each group.

7. BENTHIC EFFECTS EVALUATION

The benthic effects evaluation involves whole sediment toxicity and bioaccumulation testing of both the dredging site and reference sediments. The general procedures for Tier III toxicity tests are described in Section 11.2 of the Green Book and the ITM, and in the freshwater methods manual (EPA, 2000). Tier III bioaccumulation tests are described in Section 12.1 of the Green Book and the ITM, and in the freshwater methods manual. Reference and control sediments (if applicable) are tested in the same way as the dredged material proposed for disposal.

7.1 Tier III - Whole Sediment Toxicity Tests

The purpose of the 10 day sediment toxicity tests is to determine whether the sum of the sediment contaminants in combination with the physical characteristics will elicit a toxic response to exposed organisms after the material is deposited into the marine environment.

For projects proposing marine and estuarine disposal, two test species of those listed in the Toxicity section of Table 7 are required – one of the three marine amphipod species (depending on salinity and grain size) and the mysid shrimp. Currently only one species is required for projects proposing freshwater disposal. Species-specific test conditions are listed in Appendix VI, and in the ITM. Details are provided in EPA (1994a) for estuarine/marine amphipods, in EPA (1991b) for mysid shrimp and in Chapters 11 and 12 of the freshwater methods manual (EPA, 2000) for freshwater amphipods and midge fly larvae. All tests are static non-renewal, with the exception that renewal is allowed to control for ammonia toxicity (see below).

General guidance for the collection, handling and storage of sediments for biological testing are described in Chapter 4 of this manual and Chapter 8 of the Green Book and the ITM. Chapter 8 of the EPA amphipod test manual (EPA 1994a) must be consulted for specific guidance related to the amphipod sediment toxicity tests. The CENAE will decide whether compositing of sediment samples is permissible (see Section 4.2 of this manual).

Specific guidance on procedures for setting up, performing and breaking down the test is provided in EPA (1994a) for the amphipod species, and EPA (1991b) for the mysid species. All sediments tested may be press-sieved (determined on a case-by-case basis by visual observation) with a 1 or 2 mm sieve to remove unwanted debris and predators before being added to the test chambers. All data should be reported on the forms supplied in EPA (1994a; Appendix A, Figures A1-A5) or a close facsimile. In addition to the parameters on the forms, all observations on mortality, the formation of tubes or burrows, amphipod emergence from sediment, and any physical or behavioral abnormalities must be recorded.

Sediment chemistry for the project specific contaminants of concern, TOC and grain size analyses may be required by CENAE on subsamples of the sediments that are biologically tested. Subsamples of the dredged material, and reference and control sediments used in the test must be archived for possible future bulk analysis if the CENAE and EPA deem it necessary.

Because amphipods and mysid shrimp are sensitive to sediment ammonia, renewals of overlying

water are allowed to reduce exposure. Excessive ammonia concentrations may cause mortalities in these species and confound the mortality endpoint of interest to the dredging regulatory program, which focuses on more persistent contaminants. Ammonia toxicity changes as ephemeral environmental conditions, such as temperature, salinity, oxidation state and pH, change. To account for this potential false positive, the EPA and Corps have devised methods to reduce ammonia toxicity before any test begins [Sections 11.4.5 -11.4.5.3 of the EPA amphipod manual (EPA 1994a), as amended by the "Errata" sheet for pages 80-82 of that document]. The applicant must seek approval from the CENAE and EPA on project-specific procedures for any sediments requiring treatment for ammonia toxicity.

For the amphipod tests, to avoid toxicity from ammonia, the applicant must insure that the sediment pore water total ammonia and un-ionized ammonia concentrations are below 20 mg/l and 0.4 mg/l, respectively, for 24 hours before amphipods are added to the test chambers and during the test. Ammonia levels can be reduced by sufficiently aerating the sample and replacing two volumes of water per day (EPA 1994a). Ammonia measurements should be made in surrogate (or "dummy") test chambers set up specifically for pore water collection. Recommended procedures to set up "dummy" chambers, collect pore water and analyze for ammonia are described in Appendix VII. Total ammonia levels must be monitored in the pore water on days 1, 3 (or 5) and 10 during the test. Un-ionized ammonia can be calculated from total ammonia based on additional measurements of pH, temperature and salinity.

For the mysid shrimp test, the applicant must follow the guidance in the June 14, 1994 memo to Mario Del Vicario from Elizabeth Southerland (Appendix VIII). Here, the concern is un-ionized ammonia in the overlying water (1 cm above the sediments). The applicant must insure that the water concentrations are below 0.6 mg/L in tests run at pH of 7.9-8.0 or 0.3 mg/L at pH of 7.5 before any animals are added to the test chambers. In this case overlying water is monitored each day. The overlying water should be replaced two times per day until the levels are below the acceptable thresholds.

An alternative approach to remove ammonia is to perform a thin layer purging technique as conducted by the EPA Region 2 Environmental Laboratory (Ferretti et al., 2000). Contact CENAE for further information on this approach.

7.2 Tier III - Bioaccumulation Testing

Bioaccumulation tests provide a measure of exposure of deposit-feeding marine animals to bioavailable sediment contaminants. In this case, representatives of a bivalve and a polychaete worm species are exposed for a 28 day period to dredging site, reference and control sediments. To clarify recommendations in the Green Book, the 28 day exposure test is required for organic contaminants of concern as well as for metals. General technical guidance is provided in Section 12.1 of the ITM, Chapter 13 of the freshwater methods manual (EPA, 2000) for freshwater disposal and Lee et al. (1989), as cited in the former documents.

The two required species for marine/estuarine disposal are listed in the Bioaccumulation section of Table 7 – the sandworm, *Nereis virens*, and the bivalve *Macoma nasuta* or *Macoma balthica*.

Each species must be exposed in separate aquaria because of the predatory behavior of *Nereis virens*. It should be noted that use of another set of aquaria will require a proportionally greater amount of sediments to be collected and processed. For freshwater disposal, the oligochaete, *Lumbriculus variegatus* is used.

All aquaria must have a sediment depth of at least 5 cm. At least 20 specimens of each species are required in each test chamber, although more may be necessary to conduct the prescribed tissue analyses at the end of the test exposure. It is the applicant's responsibility to insure that the laboratory provides enough animal tissue (size and number) to run subsequent chemical analyses. Generally, it is desirable to produce 50 g (wet weight) for each replicate and species. The number of animals and the size of the aquarium will vary with the size of individual animals acquired for the test. For the species in Table 7, tissue/sediment loading should not exceed 1 g tissue (wet weight minus shell) to 50 g sediment (wet weight) (H. Lee, EPA Newport Lab, personal communication). If dioxin/furan levels are required, then a separate set of aquaria may be required to provide adequate tissue for analyses to achieve the required RLs.

Those constituents generally requiring analysis are listed in Tables 8 and 9, but may include other contaminants as determined by the Tier I review and/or chemical testing of the sediments. The final decision on which project-specific contaminants are required is made by the CENAE in consultation with other federal and state regulatory agencies. Recommended tissue extraction and analytical methods are provided in NOAA (1993), EPA/USACE (1995) and EPA (1993). The applicant must insure the contracted laboratory can reasonably achieve the required RLs listed in Tables 8 and 9 and Appendix I, if applicable. The sample preparation methods for animal tissue described in EPA (1993) and EPA/USACE (1995) are highly recommended. As mentioned above, 50 grams of tissue (wet) per replicate is recommended (or enough to obtain acceptable RLs). In addition to the contaminants, the lipids of each clam and worm tissue replicate should be analyzed using a modified Bligh and Dyer (1959) method developed by the U.S. EPA Narragansett Laboratory (EPA/AED, 1995). A copy of this method is included as Appendix IX of this document. Percent water, solids and lipid must be reported for each species and replicate.

All appropriate QA/QC measures listed in Chapters 9 and 12 of the ITM and the QA/QC manual must be followed. Tissues of organisms randomly selected prior to initiation of bioaccumulation testing (pre-test analyses) must be analyzed and reported for all contaminants analyzed in the exposed organisms. A subsample of these pre-test samples of tissue from each species must be archived as the applicant may be required to analyze this tissue at a later date for specified contaminants.

As with toxicity tests, daily records must be kept of salinity, temperature, DO, pH, flow rate, obvious mortalities and any sublethal effects. Failure of organisms to burrow into the sediment or any other physical or behavioral abnormalities must also be recorded. All bivalves (whether pre- or post-test) must be depurated for 24 hours in clean seawater prior to freezing. The polychaete, *N. virens*, must also be depurated in clean seawater (or seawater with clean sand).

7.3 Quality Control Measures

The applicant must submit documentation of all QC measures performed during analysis of the samples using the Quality Control (QC) Summary Tables in Appendix II. *If any of the control limit criteria are exceeded, the data may not be accepted.* The following additional analytical QC measures must be performed for the above referenced methods.

(a) Whole Sediment Toxicity Tests: All marine/estuarine bioassays must be performed under the conditions specified in each of the test species sheets in Appendix VI in either natural seawater or a synthetic seawater adjusted to salinity appropriate for the test species and disposal site (generally 25 to 30 ppt). Adherence with the applicable test acceptability requirements must be documented for *Ampelisca abdita*, *Eohaustorius estuarius* and *Leptocheirus plumulosis* (EPA, 1994a) and for *Hyalella azteca* (EPA, 2000).

The mean mortality of five replicates in the control sediments must be less than or equal to 10% for the test to be valid. If the control mortality is greater than 10%, the test should be repeated, or the applicant should contact the CENAE project manager for further guidance.

(b) Bioaccumulation tests - mortality: The QA/QC procedures cited in the ITM and in the freshwater methods manual (EPA, 2000) must be followed and documented for bioaccumulation testing.

Where control mortality is greater than 10% for sediment bioaccumulation samples the applicant should contact the CENAE project manager and determine whether the following conditions exist: a) adequate number of replicates to obtain statistical power; b) stressed organisms; c) contaminated control sediment; d) contamination of test system; e) quality control problems; and f) adequate tissue for chemical analyses.

(c) *Bioaccumulation tests - tissue chemistry:* In the bioaccumulation testing, the following QC checks are required for chemical analyses of tissues:

- Initial calibration
- Calculation of MDLs
- Blind analysis of spiked or performance evaluation material for calibration verification
- Continuing calibration checks
- Analysis of SRMs or LCSs
- Method Blank
- Matrix Spike
- Matrix Spike Duplicate
- Analytical replicates
- Surrogates
- Internal standards

All QA/QC for Dioxin/Furan analyses (listed in Appendix I-1) must be documented according to

the methods described in EPA Method 1613.

(c) Detection and Reporting Limits: The Method Detection and Reporting limits used in this manual are defined in Section 5.2, and the Reporting Limits for tissue chemistry are listed in Tables 8 and 9.

7.4 Statistical Analysis

Toxicity and bioaccumulation data should be analyzed as indicated in Appendix D of the ITM (summarized in Table 9). As discussed in Appendix D, these methods are described in many popular general statistics texts such as Winer (1971), Steel and Torrie (1980), Sokal and Rohlf (1981), Dixon and Massey (1983), Zar (1984) and Snedecor and Cochrane (1989). In addition, Conover (1980) is recommended for nonparametric tests. Most of these tests are included in commercially available statistics software packages. Relative to detection levels, all nondetected analytes must be reported as one half the method detection level (MDL). Results below the Reporting Limits should be reported in full as estimated, and qualified with a "J".

7.5 Data Reporting

All toxicity and bioaccumulation data (in wet weight) must be submitted to CENAE electronically and as hard copy. The required format for the electronic submission will be provided to the applicant when the SAP is approved, and is also available on the CENAE website (<www.nae.usace.army.mil/reg/rim.htm>). In addition, the applicant must provide completed Quality Control (QC) Summary Tables (Appendix II, also available on this website) and results of the QC analyses both as hard copy. This format is necessary to facilitate the project review process and to ensure completeness of the submittal. *Project data not submitted in the described formats will be considered incomplete and a resubmittal will be required*.

The applicants may submit their own data summaries and analyses; however, they must also submit the original data and copies of sampling logs so that CENAE and EPA can conduct independent analyses. All submitted data must be clearly presented and traceable to the original samples and subsamples. *Suitability determinations will not be issued based on an applicant's data analysis alone.*

TABLE 7. Organisms required for the whole sediment toxicity and bioaccumulation tests

TOXICITY 10 days

| <u>Group</u> | <u>o/Taxa</u> | <u>Habitat</u> | <u>Scientific Name</u> |
|--------------|-----------------------------|---|--|
| 1 | Amphipods ¹ | | |
| | | Marine/Estuarine and fine grain Estuarine Marine/Estuarine and coarse grain Freshwater | Ampelisca abdita Leptocheirus plumulosus Eohaustorius estuarius Hyalella azteca |
| 2 | Non-amphipods | | |
| | Mysid shrimp Midge larva | Marine/Estuarine Freshwater | Americamysis bahia Chironomus tentans |
| BIOA | CCUMULATION 28 | days | |
| <u>Group</u> | o/Taxa | <u>Habitat</u> | <u>Scientific Name</u> |
| | Bivalve | Marine/Estuarine | Macoma nasuta or M. balthica |
| | Polychaete | Marine/Estuarine | Nereis virens |
| | Oligochaete ² | Freshwater | Lumbriculus variegatus |

1

One amphipod species is required and should be selected based on disposal site conditions. Only one freshwater bioaccumulation test species is available and required for freshwater tests. 2

TABLE 8. Tissue properties, metal contaminants of concern, recommended analytical methods, and reporting limits routinely used for bioaccumulation evaluations

| <u>Contaminant</u> | Analytical <u>Method(s)</u> | Reporting <u>Limit</u> |
|---|--|---|
| Total Lipids | EPA, 1995c | 0.1% |
| Total Water Content | EPA,1986; EPA, 1987 | 0.1% |
| Metals | | ppm (wet weight) ² |
| Arsenic Cadmium Chromium Copper Lead Mercury Nickel | 200.8, 7061 200.8, 7131A 200.8, 7191 200.8, 7211 200.8, 7421 7471 200.8, 6010A | $\begin{array}{c} 0.5 \\ 0.1 \\ 1.0 \\ 1.0 \\ 1.0 \\ 0.02 \\ 1.0 \end{array}$ |
| Zinc | 200.8, 7950 | 1.0 |

TABLE 9. Organic contaminants of concern, recommended analytical methods, and reporting limits routinely used for bioaccumulation evaluations

| <u>Contaminant</u> | Analytical <u>Method(s)¹</u> | Reporting <u>Limit (wet weight)</u> |
|---|---|--|
| PAHs | 1625C, 8270C, 8100 NOAA, 1993 ² | 20 ppb ² |
| Acenaphthene Acenaphthylene Anthracene Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenzo(a,h)anthracene Fluoranthene Fluorene Indeno(1,2,3-cd)pyrene Naphthalene Phenanthrene Pyrene | | |
| Pesticides | 8081B ² | 1 ppb ² |
| Aldrin cis- and trans-Chlordane cis- and trans-Nonachlor Oxychlordane 4,4'-DDT, DDE, DDD Dieldrin alpha- and beta-Endosulfan Endrin Heptachlor Heptachlor Heptachlor epoxide Hexachlorobenzene Lindane Methoxychlor | | |
| Toxaphene | | 50 ppb |

 TABLE 9 (continued). Organic contaminants of concern, recommended analytical methods, and reporting limits routinely used for bioaccumulation evaluations

| <u>Contaminant</u> | | Analytical <u>Method(s)¹</u> | Reporting <u>Limit (wet weight)</u> |
|--------------------|----------------------------|--|--|
| PCB Congene | ers ³ | 8082A ² | 0.5 ppb ² |
| 8 | 2,4' diCB | | |
| 18 | 2,2',5 triCB | | |
| 28 | 2,4,4' triCB | | |
| 44 | 2,2',3,5' tetraCB | | |
| 52 | 2,2',5,5' tetraCB | | |
| 66 | 2,3',4,4' tetraCB | | |
| 101 | 2,2',4,5,5' pentaCB | | |
| 105 | 2,3,3',4,4' pentaCB | | |
| 118 | 2,3',4,4',5 pentaCB | | |
| 128 | 2,2',3,3',4,4' hexaCB | | |
| 138 | 2,2',3,4,4',5' hexaCB | | |
| 153 | 2,2',4,4',5,5' hexaCB | | |
| 170 | 2,2',3,3',4,4',5 heptaC | В | |
| 180 | 2,2',3,4,4',5,5' heptaC | В | |
| 187 | 2,2',3,4',5,5',6 heptaC | В | |
| 195 | 2,2',3,3',4,4',5,6 octaC | СВ | |
| 206 | 2,2',3,3',4,4',5,5',6 nor | naCB | |
| 209 | 2,2',3,3',4,4',5,5',6,6' d | lecaCB | |

- 1 The specified methods are recommendations only. Other acceptable methodologies capable of meeting the TQLs may be used. Sample preparation methodology (e.g. extraction and cleanup) and sample size may need to be modified to achieve the required target quantitation limits.
- 2 Applies to each analyte listed below unless otherwise noted.
- 3 Total PCBs are to be estimated based on the following: Total = 2 X [sum of 18 NOAA summation congeners listed above] (T. Wade, personal communication). For values below the MDL, use one half the MDL; for values between the MDL and the RL, use estimated values.

TABLE 10. Recommended statistical methods for biological testing¹

| <u>Statistic</u> | Method |
|----------------------|--|
| Normality | Shapiro-Wilk's Test Kolmogorov-Smirnov (K-S) Test Normality tests found in SYSTAT or SPSS |
| Equality of Variance | Bartlett's Test (should not be used to test equality of ranks) Levene's Test F_{max} Test Cochran's Test |
| Parametric | Fisher's Least Significant Difference (LSD) (if raw or transformed are normally distributed) in conjunction with analysis of variance (ANOVA). |
| Nonparametric | LSD on rankits (= van der Waerden's Test in Conover, 1980) (if the data converted to rankits are found to be normally distributed); or Conover T-Test (Conover, 1980) (if the variances of the ranks are not significantly different); or |
| | One tailed T-Test for unequal variances for each pair of treatments (if the ranks are significantly unequal). |

1 Summarized from Appendix D (EPA/USACE, 1998)

8. REFERENCES

Allen, H.E., F. Gongmin, W. Boothman, D. DiToro and J.D. Mahony. 1991. Determination of Acid Volatile Sulfides and Simultaneously Extractable Metals in Sediment. U.S. Environmental Protection Agency, Office of Water, Washington D.C., Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment, August 1991.

Ankley, G.T., G.J. Niemi, K.B. Lodge, H.J. Harris, D.L. Beaver, D.E. Tillit, T.R. Schwartz, J.P. Giesy, P.D. Jones and C. Hagley. 1993. Uptake of Planar Polychlorinated Biphenyls and 2,3,7,8-Substituted Polychlorinated Dibenzofurans and Dibenzo-p-dioxins by Birds Nesting in the Lower Fox River and Green Bay, Wisconsin, USA, Arch. Environ. Contam. Toxicol. 24: 332-344.

APHA. 1995. Standard Methods for the Analysis of Water and Waste Water. 19th ed. American Public Health Association, American Water Works Association, Water Pollution Control Federation, Washington, DC.

ASTM. 1998a. Standard Methods for Particle-Size Analysis of Soils. In 1998 Annual Book of ASTM Standards Vol. 4.08. Philadelphia, PA. (D 422-63, D421-85, D2217-85).

ASTM. 1998b. Standard Test Method for Specific Gravity of Soils. In 2001 Annual Book of ASTM Standards Philadelphia, PA. (D 854-92).

ASTM. 1998c. Standard Test Method for Liquid limit, Plastic Limit and Plasticity Index of Soils In 2001 Annual Book of ASTM Standards Vol. 4.08. Philadelphia, PA. (D 4318-95).

ASTM. 1998d. Standard Guide for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians. In 2001 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.04, Philadelphia, PA. (E 729-88a).

ASTM. 1998e. Standard Guide for Conducting Acute Toxicity Tests on Aqueous Effluents with Macroinvertebrates, and Amphibians In 2001 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.04, Philadelphia, PA. (E 1192-88).

ASTM. 1998f. Standard Guide for Conducting Static Acute Toxicity Tests Starting with Embryos of Four Species of Saltwater Bivalve Molluscs In 2001 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.04, Philadelphia, PA. (E 724-94).

ASTM. 1998g. Standard Guide for Conducting Acute Toxicity Tests with Echinoid larvae In 2001 Annual Book of ASTM Standards Vol. 11.05, Philadelphia, PA. (E 1563-95).

Bligh, E.G. and W.J. Dyer. 1959. A rapid method of total lipid extraction and purification. Can. J. Biochem. Physiol. 37: 911-917.

Bloom, N.S. and E.A. Crecelius. 1983. Determination of Mercury in Seawater at Sub-Nanogram Per Liter Levels. Marine Chem.14: 49-59.

Burgess, R.M. 1995. US EPA Environmental Research Lab, Narragansett, RI. Telephone conversation with D. Tomey.

Conover, W.J. 1980. Practical Nonparametric Statistics. 2nd Ed. John Wiley & Sons, New York, NY 493 pp.

Dixon, W.J. and F.J. Massey. 1983. Introduction to Statistical Analysis. 4th Ed. MaGraw-Hill Book Co., New York, NY. 678 pp.

DOA. 1980. Laboratory Soils Testing. Engineering and Design, Engineer Manual EM1110-2-1906, rev. to 1970 publ., Appendix II. HQ Dept. of Army Office of Chief of Engineers.

EPA. 1979. Revised 1983. Methods for the Chemical Analysis of Water and Wastes. EPA-600/4-79-020. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH.

EPA. 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. EPA-600/4-83-004. Prepared by Environmental Protection Agency, Office of Research and Development, Monitoring Systems and Quality Assurance Branch, Washington, DC. 29 pp.

EPA. 1984. Guidance for the Preparation of Combined/Work Quality Assurance Project Plan for Environmental Monitoring. Environmental Protection Agency, Office of Water Regulations and Standards (OWRS) QA-1, Washington, DC.

EPA. 1986. Test Methods for Evaluating Solid Waste. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, DC.

EPA. 1987. Quality Assurance/Quality Control (QA/QC) for 301(h) Monitoring Program: Guidance on Field and Laboratory Methods. EPA 430/9-86-004. NTIS Number PB 87-221164. Prepared for the Environmental Protection Agency Office of Marine and Estuarine Protection by Tetra Tech, Inc., Bellevue, WA.

EPA. 1989. Preparing Perfect Project Plans; A Pocket Guide for the Preparation of Quality Assurance Project Plans. EPA/600/9-89/087. Prepared by Risk Reduction Engineering Laboratory, Cincinnati, OH. October 1989. 62 pp.

EPA. 1991a. Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch.

EPA. 1991b. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 4th ed. EPA600/4-90/027. Office of Research and Development, Washington, D.C. 20460.

EPA. 1992. Determination of Total Organic Carbon in Sediment. Environmental Protection Agency Region II, Environmental Services Division, Monitoring Management Branch, Edison, NJ.

EPA. 1993. Recommended Analytical Techniques and Quality Assurance/Quality Control Guidelines for the Measurement of Organic and Inorganic Analytes in Marine Sediments and Tissue Samples. Draft, Prepared by US EPA Environmental Research Laboratory, Narragansett, RI. 83 pp.

EPA. 1994a. Methods for Assessing the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Amphipods. U.S. Environmental Protection Agency. Office of Research and Development. Washington D.C. EPA/600/R-94/025.

EPA. 1994b. Short-term Methods for Measuring Chronic Toxicity of Effluents and surface waters to Marine and Estuarine Organisms. Second Ed. U.S. Environmental Protection Agency. Office of Research and Development, Cincinnati, OH. 341 pp. EPA/600/4-91-003.

EPA. 1994c. EPA Requirements for Quality Assurance Project Plans. Current Draft Version: August 1994. U.S. Environmental Protection Agency, Quality Assurance Division, Washington D.C. EPA QA/R-5.

EPA. 1995a. Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. U.S. Environmental Protection Agency, Washington D.C. EPA 821-R-95-034.

EPA. 1995b. Method 1632: Determination of Inorganic Arsenic Trace Elements in Water by Hydride Generation Flame Atomic Absorption. U.S. Environmental Protection Agency, Washington D.C. April 1995, Draft EPA 821-R-96-028.

EPA. 1995c. Method 1636: Determination of Hexavalent Chromium by Ion Chromatography. U.S. Environmental Protection Agency, Washington D.C. April 1995, Draft EPA 821-R-96-029.

EPA. 1996a. Method 1631: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atom Fluorescence Spectrometry. U.S. Environmental Protection Agency, Washington D.C. January 1996, Draft EPA 821-R-96-001.

EPA. 1996b. Method 1637: Determination of Trace Elements in Ambient Waters by Chelation Preconcentration with Graphite Furnace Atomic Absorption. U.S. Environmental Protection Agency, Washington D.C. January 1996, Draft EPA 821-R-96-004.

EPA. 1996c. Method 1639: Determination of Trace Elements in Ambient Waters by Stabilized Temperature Graphite Furnace Atomic Absorption. U.S. Environmental Protection Agency, Washington D.C. January 1996, Draft EPA 821-R-96-006.

EPA. 1996d. Method 1640: Determination of Trace Elements in Ambient Waters by On-line Chelation Preconcentration and Inductively coupled Plasma-Mass Spectrometry. U.S.

Environmental Protection Agency, Washington D.C. January 1996, Draft EPA 821-R-96-007.

EPA. 1998. EPA Guidance for Quality Assurance Project Plans, Final: February 1998. U.S. Environmental Protection Agency, Quality Assurance Division, Office of Research & Development, Washington, D.C. EPA QA/G-5.

EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. Second Edition, dated March 2000. U.S. Environmental Protection Agency, Office of Research and Development, Washington D.C. EPA/600/R-99/-064.

EPA. 2001a. Appendix A, Method 608 -- Organochlorine Pesticides and PCBs. 625. Title 40 Code of Federal Regulations, Part 136.

EPA. 2001b. Appendix A, Method 625 -- Base/Neutrals and Acids. Title 40 Code of Federal Regulations, Part 136.

EPA. 2001c. Appendix B to Part 136 -- Definition and Procedure for the Determination of the Method Detection Limit. Revision 1.11. Title 40 Code of Federal Regulations, Part 136.

EPA. 2001d. Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual (EPA-823-B-01- 002) October 2001. EPA, Standards and Health Protection Division (4305), Office of Science and Technology, Washington, DC.

EPA. 2001e. EPA Requirements for Quality Assurance Project Plans, Final: March 2001. U.S. Environmental Protection Agency, Quality Staff, Office of Environmental Information, Washington, D.C. EPA QA/R-5.

EPA/AED. 1995. AED Laboratory Operation Procedure Measurement of Total Lipids using Modification Bligh-Dyer Method. Dated March 15, 1995. U.S. Environmental Protection Agency. Atlantic Ecology Division. Narragansett, RI.

EPA/AED. 1996. AED Laboratory Operating Procedure Conducting the Sea Urchin Larval Development Test. AED LOP 1.03.007 Revision 1, November, 1996. U.S. Environmental Protection Agency. Atlantic Ecology Division. Narragansett, RI. 9pp.

EPA/USACE. 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). Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material, Environmental Effects Laboratory, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS. 2nd printing 1978.

EPA/USACE. 1991. Evaluation of Dredged Material for Ocean Disposal (Testing Manual).

Environmental Protection Agency/U.S. Army Corps of Engineers. U.S. Army Engineer Waters Experiment Station, Vicksburg, MS.

EPA/USACE. 1995. QA/QC Guidance for Sampling and Analysis of Sediments, Water and Tissue for Dredged Material Evaluations Chemical Evaluations. Environmental Protection Agency/U.S. Army Corps of Engineers. U.S. Environmental Protection Agency, Office of Water, Washington D.C. EPA 823-B-95-001.

EPA/USACE. 1998. Evaluation of Dredged Material for Proposed for Discharge in Waters of the U.S. - Testing Manual, Inland Testing Manual. U.S. Environmental Protection Agency, Office of Water, Washington D.C.

EPA Region 1/USACE New England Division. 1989. Guidance for Performing Tests on Dredged Material To Be Disposed of in Open Waters. Environmental Protection Agency, Region I, Boston, MA/U.S. Army Corps of Engineers, New England Division, Waltham, MA. 32 pp.

Ferretti, J.A. D.F. Calesso, and T.R. Hermon. 2000. Evaluation of Methods to Removce Ammonia Interference in Marine Sediment Toxicity Tests. Environmental Toxicology and Chemistry 19(8): 1935-1941.

Klute, A. (ed) 1986. Methods of Soil Analysis Part I. Physical and Mineralogical Methods 2nd ed. American Society of Agronomy, pp 363-375.

Lee, H. III, B.L. Boese, J. Peltier, M. Sinsor, D.T. Sprecht and R.C. Randall. 1989. Guidance Manual: Bedded Bioaccumulation Tests. ERL-N Contribution No. N111, EPA 600/X-89/302. Newport, OR.

Lee, H. III. 1995. US EPA Environmental Research Lab, Newport, OR. Telephone conversation with D. Tomey.

McFarland, V.A. and J.U. Clarke. 1989. Environmental occurrence, abundance, and potential toxicity of polychlorinated biphenyl congeners: considerations for a congener-specific analysis. Environmental Health Perspectives 81: 225-239.

NOAA. 1991. Second Summary of Data on Chemical Contaminants in Sediments from the National Status and Trends Program. NOAA Technical Memo. NOS OMA 59. U.S. Dept. Commerce, NOAA National Ocean Service, Rockville, MD.

NOAA. 1993. Standard Analytical Procedures of the NOAA National Analytical Facility. NOAA Tech. Mem. NMFS F/NWC-92, 1986-89. National Status and Trends Program, National Oceanic and Atmospheric Administration, NOAA N/OMA32, 11400 Rockville Pike, Rockville, MD 20852. 3rd ed.

NYDEC. 1991. Analytical Method for the Determination of PCB Congeners by Fused Silica Capillary Column Gas Chromatography with Electron Capture Detector. NYSDEC #91-11.

Available from Larry Bailey, New York State Department of Environmental Conservation, 50 Wolf Road, Albany, NY 12233, Phone 518-457-7471.

Plumb, R.H., Jr. 1981. Procedure for Handling and Chemical Analysis of Sediment and Water Samples. Tech. Rep. EPA/CE-81-1. Prepared by Great Lakes Laboratory, State University College at Buffalo, Buffalo, NY, for the Environmental Protection Agency/U.S. Army Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

Pruell, Richard. 1995. US EPA Environmental Research Lab, Narragansett, RI. Telephone conversation with D. Tomey.

Puget Sound Estuary Program (PSEP). 1986. Total Organic Carbon (TOC). Pages 23-26 In Recommended Protocols for Measuring Conventional Sediment Variables in Puget Sound. Prepared for U.S. Environmental Protection Agency, Region 10, Seattle, WA by Tetra Tech, Inc. Bellevue WA.

Rice, C.D., F.A. Espourteille, and R.J.Hugget. 1987. Analysis of Tributyltin in Estuarine Sediments and Oyster Tissue, Crassostrea virginica. Appl. Organomet. Chem. 1:541-544.

Schwartz, T.R., D.E. Tillit, K.P. Feltz, and P.H. Peterman. 1993. Determination of Mono- and Non-o,o'-Chlorine Substituted polychlorinated Biphenyls in Aroclors and Environmental Samples, Chemosphere 26(8):1443-1460.

Seriano J.L., A.M. El-Husseini and T.L. Wade. 1991. Isolation of Planar Polychlorinated Biphenyls by Carbon Column Chromatography, Chemosphere 23(7): 915-924.

Snedecor, G.W. and G.C.Cochrane. 1989. Statistical Methods. 8th Ed. The Iowa State University Press, Ames, IA 507 pp.

Sokal, R.R. and F.J. Rohlf. 1981. Biometry. 2nd Ed. W.H. Freeman and Company, San Francisco, CA 859 pp.

Steel, R.G.D. and J.H. Torrie. 1980. Principles and procedures of Statistics. 2nd Ed. McGraw-Hill Company, New York, NY 633 pp.

Ulher, A.D. and G.S. Durrel. 1989. Measurement of Butyltin Species in Sediments by n-pentyl Derivation with Gas Chromatography/Flame Photometric Detection (GC/FPD) Battelle Ocean Sciences Project N-0519-6100, Duxbury, MA.

Wade, T. 1996. Geochemical and Environmental Research Group, Texas A & M University, College Station, TX. Telephone conversation with D. Tomey.

Winer, B.J. 1971. Statistical Principles in Experimental Design. 2nd Ed. McGraw-Hill Book Company, New York, NY 907 pp.

Zar. J.H. 1984. Biostatistical Analysis. 2nd Ed. Prentice-Hall, Inc., Englewood Cliffs, NJ 717 pp.