



United States Army Corps of Engineers
New York District

Environmental Protection Agency
Region 2

# FINAL

# GUIDANCE FOR PERFORMING TESTS ON DREDGED MATERIAL PROPOSED FOR OCEAN DISPOSAL

April 2016

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## LIST OF ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| DA | Department of the Army |
| DDD | dichlorodiphenyldichloroethane |
| DDE | dichlorodiphenyldichloroethylene |
| DDT | dichlorodiphenyltrichloroethane |
| DOC | demonstration of capability |
| $EC_{50}$ | median effective concentration |
| EPA | United States Environmental Protection Agency |
| HARS | Historic Area Remediation Site |
| HDPE | high density polyethylene |
| HMW | high molecular weight |
| HpCDD | heptachlorodibenzo-p-dioxin |
| HpCDF | heptachlorodibenzofuran |
| HxCDD | hexachlorodibenzo-p-dioxin |
| HxCDF | hexachlorodibenzofuran |
| $LC_{50}$ | median lethal concentration |
| LMW | low molecular weight |
| LPC | Limiting Permissible Concentration |
| MPRSA | Marine Protection, Research, and Sanctuaries Act of 1972 |
| NYD | New York District, U.S. Army Corps of Engineers |
| OCDD | octachlorodibenzo-p-dioxin |
| OCDF | octachlorodibenzofuran |
| OMB | Office of Management and Budget |
| PAH | polycyclic aromatic hydrocarbon |
| PCB | polychlorinated biphenyl |
| PeCDD | pentachlorodibenzo-p-dioxin |
| PeCDF | pentachlorodibenzofuran |
| QA | quality assurance |
| QC | quality control |
| RL | reporting limit |
| SOP | standard operating procedure |

| | |
|---|---|
| SRM | standard reference material |
| TCDD | tetrachlorodibenzo-p-dioxin |
| TCDF | tetrachlorodibenzofuran |
| TOC | total organic carbon |
| USACE | U.S. Army Corps of Engineers |
| WQC | water quality criteria |

# 1.0 INTRODUCTION

## 1.1 Purpose

This guidance document presents the sediment testing guidelines and requirements to be used by applicants who wish to obtain a Department of the Army (DA) permit from the New York District (NYD) of the United States Army Corps of Engineers (USACE) for dredging and placement of dredged material at the Historic Area Remediation Site (HARS) in the Atlantic Ocean. The USACE and Environmental Protection Agency (EPA) are responsible for reviewing test results for compliance with the Ocean Dumping Criteria as described below.

This manual and any subsequent updates can be downloaded at www.nan.usace.army.mil.

## 1.2 Background

Section 103 of the Marine Protection, Research, and Sanctuaries Act of 1972 (MPRSA), Public Law 92-532, requires that all proposed operations involving the transportation and discharge of dredged material into ocean waters be evaluated to determine the potential environmental impact of such activities. The proposed ocean placement must be evaluated through the use of criteria published by the Environmental Protection Agency (EPA) in Title 40 of the Code of Federal Regulations, Parts 220-228 (40 CFR 220-228) (hereafter: the Regulations). In accordance with Subsection 227.27(b) of the Regulations, EPA and USACE developed a testing manual to define procedures for evaluating the suitability of dredged material for ocean disposal that are based upon the biological testing requirements of the Regulations. The first testing manual, *Ecological Evaluation of Proposed Discharge of Dredged Material into Ocean Waters*, commonly referred to as the "Green Book," was published by EPA and USACE in 1977. The 1977 Green Book was updated in 1991 and re-titled *Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual)* (EPA/USACE, 1991). Copies of the national guidance may be obtained as an Adobe Acrobat (i.e., pdf) file at: http://water.epa.gov/type/oceb/oceandumping/dredgedmaterial/gbook_index.cfm
This regional manual provides instructions for implementing the broad technical guidance contained in the Green Book providing regional specifications such as: the acceptable species for use in biological tests; the identification of contaminants of regional concern; Region-specific quality assurance requirements; and other methodologies that reflect the exposures and receptors that are appropriate for regional sediment assessments. This manual updates and replaces the existing regional implementation manual entitled *Guidance for Performing Tests on Dredged Material to be Disposed of in Ocean Waters* (USACE NYD/EPA Region 2, 1992).

To keep up with technical advances and new procedures in the assessment of sediment contamination and potential impacts due to dredging and disposal activity, sections of this document may be revised or updated. When major revisions or new tests are instituted, revised or new sections will be made available for download on the NYD website (www.nan.usace.army.mil). To receive notifications of available revisions to the testing manual, applicants and laboratories are encouraged to contact NYD to add your electronic mail contact information to the distribution list.

All inquiries on sampling and testing procedures described in this manual or requests to be placed on the distribution list for notification of revisions to the testing process should be directed to:

United States Army Corps of Engineers
New York District, CENAN-OP-SD
Dredged Material Management Section
26 Federal Plaza, Rm. 1937
New York, NY 10278-0090
(917) 790-8539

Email: oksana.s.yaremko@usace.army.mil

### 1.3 Changes from the Previous Edition

As stated above, this manual updates and replaces the *Guidance for Performing Tests on Dredged Material to be Disposed of in Ocean Waters* (USACE NYD/EPA Region 2, 1992). Changes from the previous edition are substantive and include the following:

Summary of Changes – General:

- Quality assurance plan development;
- Quality assurance and quality control for chemical analysis;
- Laboratory demonstration of capability guidelines;
- Laboratory demonstration of capability acknowledgement; and
- Overall format of the document.

Summary of Changes – Biological:

- Alternate ammonia management procedures for solid phase tests;
- Reference sediment handling procedures and numeric test acceptability criteria;
- Ammonia toxicity threshold values for *Americamysis bahia* (previously *Mysidopsis bahia)* solid phase tests revised;
- Bioaccumulation test static renewal procedures; and
- Report preparation checklists for biological data.

Summary of Changes – Chemical:

- Sample volume, container, preservation and holding times for chemical analyses;
- Quality control samples for chemical testing of tissue, site water, elutriate and sediment;
- Report preparation checklists for chemical data; and
- Chemical reporting limits.

# 2.0 REGULATORY REQUIREMENTS

## 2.1 Overview of MPRSA

The primary federal environmental statute governing transportation of dredged material for the purpose of dumping it into ocean waters (seaward of the baseline of the territorial sea) is the Marine Protection, Research, and Sanctuaries Act (MPRSA), also referred to as the Ocean Dumping Act. 33 U.S.C. § 1401 et seq.

In accordance with section 103 of the MPRSA, the USACE is the permitting authority for ocean dumping of dredged material, subject to EPA review and concurrence that the material meets applicable ocean dumping criteria at 40 CFR Parts 227 & 228. Under MPRSA section 103(d), if EPA determines the ocean dumping criteria are not met, dumping may occur only if EPA grants a waiver of the criteria.

Section 103 of the MPRSA allows the USACE to issue permits, after notice and opportunity for public hearings, for the transportation of dredged material for the purpose of dumping it into ocean waters, where the USACE determines, subject to EPA concurrence, that the dumping will not unreasonably degrade or endanger human health, welfare, or amenities, or the marine environment, ecological systems, or economic potentialities. 33 U.S.C. §§ 1413(a) & (c). This evaluation and determination are initially performed by the USACE, using criteria promulgated by EPA (see 40 CFR Parts 227 & 228). Under other authorities, the USACE also is required to consider navigation, economic and industrial development, and foreign and domestic commerce, as well as the availability of alternatives to ocean disposal. Proposed ocean disposal of dredged material also must comply with the permitting and dredging regulations in 33 CFR Parts 320-330 and 335-338.

In addition to the issuance of private permits, USACE regulations apply to Corps authorization of "federal projects involving dredged materials." Such regulations apply the same ocean dumping criteria, factors to be evaluated, and procedures and requirements that apply to the issuance of permits under MPRSA sections 103(a)-(d) and 104 (a) & (d).

## 2.2 Testing and Evaluation Under MPRSA

This section contains guidance for deciding when testing should be conducted to determine the suitability of dredged material for discharge into ocean waters regulated under section 103 of the MPRSA. There are two instances where dredged material may be determined to be suitable for placement in the ocean (i.e., use at the HARS) without having to perform the full suite of tests described in this manual. These instances are described in Sections 2.2.1 and 2.2.2, below.

### 2.2.1 Dredged Material that the Regulations Exclude from Need for Testing

The Regulations (at 40 CFR 227.13 (b)) list three conditions that qualify dredged material from a proposed project to be determined suitable for ocean disposal without undergoing the testing prescribed in this manual. Two of these "exclusionary criteria" are applicable to project materials being proposed for discharge at offshore sites such as the HARS; if a project material meets either of the two following criteria, NYD and EPA may determine the material to be suitable for placement at the HARS without further testing:

a) The proposed dredged material is composed predominantly of sand[1], gravel, rock, or any other naturally occurring bottom material with particle size larger than silt, and the material is found in areas of high current or wave energy such as streams with large bed loads or coastal areas with shifting bars and channels.
b) The material proposed for dumping is substantially the same as the substrate at the proposed disposal site prior to any dumping activities, and the proposed dredging site is far removed from known existing and historical sources of pollution so as to provide reasonable assurance that the dredged material has not been contaminated by such pollution.

Material that does not meet the above exclusionary criteria is subject to toxicity and bioaccumulation testing (see 40 CFR 227.13(c)). However, toxicity and bioaccumulation testing has already been conducted on material from certain geological deposits. Under certain circumstances (see following sub-section), the previous testing results may be applicable to the material under consideration.

### 2.2.2 Previously Characterized Proglacial Red Clay and Glacial Till

Red clay occurs as a laterally continuous, relatively thick and homogeneous sedimentary deposit throughout Newark Bay and the Kill van Kull. This clay is associated with Pleistocene glacial lakes. Due to its age, vertical position, and sedimentary and hydraulic characteristics, the red clay has minimal levels of contamination associated with it. Previous testing of these sediments, including toxicity and bioaccumulation testing (conforming to all quality assurance requirements in this manual), has determined the clay from this deposit to be HARS-suitable. NYD and Region 2 have made a programmatic determination that future dredged material that is shown to be from these deposits is likewise concluded to be suitable for placement at the HARS and is deferred from further site-specific testing.

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[1]The predominance of sand, gravel, or rock should be determined based on grain size analysis using the Unified Soil Classification System (USCS) (ASTM D 2487-06, 2006). Predominantly sand, gravel, or rock is interpreted by Region 2 and the New York District to include the USCS Clean Sands and Gravel Groups (i.e., gravels and sands having no more than 12% fines, i.e., those particles passing through a No. 200 (75-µm) sieve).

Sedimentary materials of glacial origin underlie more recently deposited sediments in northwestern portions of NY/NJ Harbor (Arthur Kill, Kill van Kull, Newark Bay, and portions of the Bayonne waterfront). Referred to as "glacial till," these materials can range from clay to boulder in size and may or may not occur as stratified and sorted deposits. Eight separate areas of this glacial unit were sampled and tested and found suitable for placement at the HARS. NYD and Region 2 have made a programmatic determination that future dredged material that is shown to be from these sediment deposits is likewise concluded to be suitable for placement at the HARS and is deferred from further site-specific testing.

Detailed protocols for acquiring the geotechnical data to support the decision that project materials are suitable for ocean placement at the HARS without further testing because they are part of the previously characterized formations are found in the NYD memorandum for distribution entitled *Standards for Submission of Geotechnical Information Used for Determination of Pleistocene Glacial Till and/or Red Clay* (Appendix A).

### 2.3 Ocean Dumping Criteria (MPRSA)

EPA's ocean dumping criteria (40 CFR Parts 227 & 228) provide the requirements for evaluating the potential for adverse effects of ocean discharge of dredged material on marine organisms and human uses of the ocean.

#### 2.3.1 Trace Contaminants

As stated in 40 CFR 227.6(a), "… the ocean dumping, or transportation for dumping, of materials containing the following constituents as other than trace contaminants will not be approved on other than an emergency basis:

- Organohalogen compounds;
- Mercury and mercury compounds;
- Cadmium and cadmium compounds;
- Oil of any kind or in any form, including but not limited to petroleum, oil sludge, oil refuse, crude oil, fuel oil, heavy diesel oil, lubricating oils, hydraulic fluids, and any mixtures containing these, transported for the purpose of dumping insofar as these are not regulated under the FWPCA;
- Known carcinogens, mutagens, or teratogens or materials suspected to be carcinogens, mutagens, or teratogens by responsible scientific opinion."

40 CFR 227.6(b) further states: "These constituents will be considered to be present as trace

contaminants only when they are present in materials otherwise acceptable for ocean dumping in such forms and amounts in liquid, suspended particulate, and solid phases that the dumping of the materials will not cause significant undesirable effects, including the possibility of danger associated with their bioaccumulation in marine organisms."

"The potential for significant undesirable effects due to the presence of these constituents shall be determined by application of results of bioassays on liquid, suspended particulate, and solid phases of dredged material according to procedures acceptable to EPA and the USACE." (40 CFR 227.6(c)). "Materials shall be deemed environmentally acceptable for ocean dumping only when the following conditions are met:

(1) The liquid phase does not contain any of these constituents in concentrations which will exceed applicable marine water quality criteria after allowance for initial mixing; provided that mercury concentrations in the disposal site, after allowance for initial mixing, may exceed the average normal ambient concentrations of mercury in ocean waters at or near the dumping site which would be present in the absence of dumping, by not more than 50 percent; and

(2) Bioassay results on the suspended particulate phase of the material do not indicate occurrence of significant mortality or significant adverse sublethal effects including bioaccumulation due to the dumping of wastes containing the constituents listed in paragraph (a) of this section. These bioassays shall be conducted with appropriate sensitive marine organisms as defined in 227.27(c) using procedures for suspended particulate phase bioassays approved by EPA, or, for dredged material, approved by EPA and the Corps of Engineers. Procedures approved for bioassays under this section will require exposure of organisms for a sufficient period of time and under appropriate conditions to provide reasonable assurance, based on consideration of the statistical significance of effects at the 95 percent confidence level, that, when the materials are dumped, no significant undesirable effects will occur due either to chronic toxicity or to bioaccumulation of the constituents listed in paragraph (a) of this section; and

(3) Bioassay results on the solid phase of the wastes do not indicate occurrence of significant mortality or significant adverse sublethal effects due to the dumping of wastes containing the constituents listed in paragraph (a) of this section. These bioassays shall be conducted with appropriate sensitive benthic marine organisms using benthic bioassay procedures approved by EPA, or, for dredged material, approved by EPA and the Corps of Engineers. Procedures approved for bioassays under this section will require exposure of organisms for a sufficient period of time to provide reasonable assurance, based on considerations of statistical significance of effects at the 95 percent confidence level, that, when the materials are dumped, no significant undesirable effects will occur due either to chronic toxicity or to bioaccumulation of the constituents listed in paragraph (a); and

(4) For persistent organohalogens not included in the applicable marine water quality criteria, bioassay results on the liquid phase of the waste show that such compounds are not present

in concentrations large enough to cause significant undesirable effects due either to chronic toxicity or to bioaccumulation in marine organisms after allowance for initial mixing."

### 2.3.2 Limiting Permissible Concentration

Dredged material may not exceed the LPC (40 CFR 227.13(c)).

As defined in 40 CFR 227.27(a), the LPC of the liquid phase of a material is that concentration of a constituent which, after allowance for initial mixing (as defined in 40 CFR 227.29), does not exceed applicable marine water quality criteria (as defined in 40 CFR 227.31). When there are no applicable marine water quality criteria or there is reason to suspect synergistic effects of certain contaminants, the LPC is that concentration of material in the receiving water which, after allowance for initial mixing, will not exceed a toxicity threshold defined as 0.01 of a concentration shown to be acutely toxic to appropriate sensitive marine organisms. When there is reasonable scientific evidence on a specific material to justify the use of an application factor other than 0.01, such alternative application factor shall be used in calculating the LPC. (40 CFR 227.27(a)(2) *&* (3)).

As defined in 40 CFR 227.27(b) *&* (c), the LPC of the suspended particulate and solid phases of a material means that concentration which will not cause:

- Unreasonable acute or chronic toxicity or other sublethal adverse effects based on bioassay results using appropriate sensitive marine organisms in the case of the suspended particulate phase, or appropriate sensitive benthic marine organisms in the case of the solid phase; and

- Accumulation of toxic materials in the human food chain.

Bioassays are to be conducted in accordance with procedures approved by EPA and the USACE. Suspended particulate phase bioaccumulation testing is not required. (40 CFR 227.27(b)).

### 2.3.3 Dredged Material Testing Described in this Manual

When dredged material proposed for ocean placement at the HARS does not meet the exclusionary criteria listed in 40 CFR 227.13(b) (see section 2.2.1), further testing of the liquid, suspended particulate, and solid phases[1] is required. Box 2-1 summarizes the regulatory requirements (in

[1] Pursuant to 40 CFR 227.32(b)(1), the liquid, suspended particulate, and solid phases of dredged material are defined as:

Liquid: The centrifuged and 0.45 micron-filtered supernatant remaining after one hour undisturbed settling of the mixture resulting from a vigorous 30-minute agitation of one part bottom sediment from the dredging site with four parts water (volume/volume) collected from the dredging or disposal site, as appropriate for the type of dredging operation.

Suspended Particulate: The supernatant obtained in the liquid phase (above) prior to centrifugation and filtering.

shaded boxes) for trace contaminants and limiting permissible concentration (40 CFR 227.13(c); discussed in sections 2.3.1 and 2.3.2), as well as the testing procedures (in ovals) required in this manual to fulfill those requirements.

Based on the results of such testing, dredged material can be considered to be environmentally acceptable for ocean dumping only under the following conditions:

- The material is in compliance with the requirements of 40 CFR 227.6 (regarding constituents that may not be present in the material as other than trace contaminants); **and**

- (i) All major constituents of the liquid phase are in compliance with the applicable marine water quality criteria, described in section 227.31, after allowance for initial mixing; or (ii) When the liquid phase contains major constituents not included in the applicable marine water quality criteria, or there is reason to suspect synergistic effects of certain contaminants, bioassays on the liquid phase of the dredged material show that it can be discharged so as not to exceed the limiting permissible concentration (LPC) as defined in paragraph (a) of section 227.27; **and**

- Bioassays on the suspended particulate and solid phases show that it can be discharged so as not to exceed the limiting permissible concentration (LPC) as defined in paragraph (b) of section 227.27.

Due to the contaminant levels historically found in NY/NJ Harbor and surrounding area, it has been determined by both EPA and NYD that all sediment, tissue, site water, and elutriate samples must be collected and analyzed for the contaminants listed in Appendix B and all biological tests must be conducted on dredged material proposed for ocean disposal that does not meet the characteristics specified in Subsections 2.2.1 and 2.2.2 of this manual..

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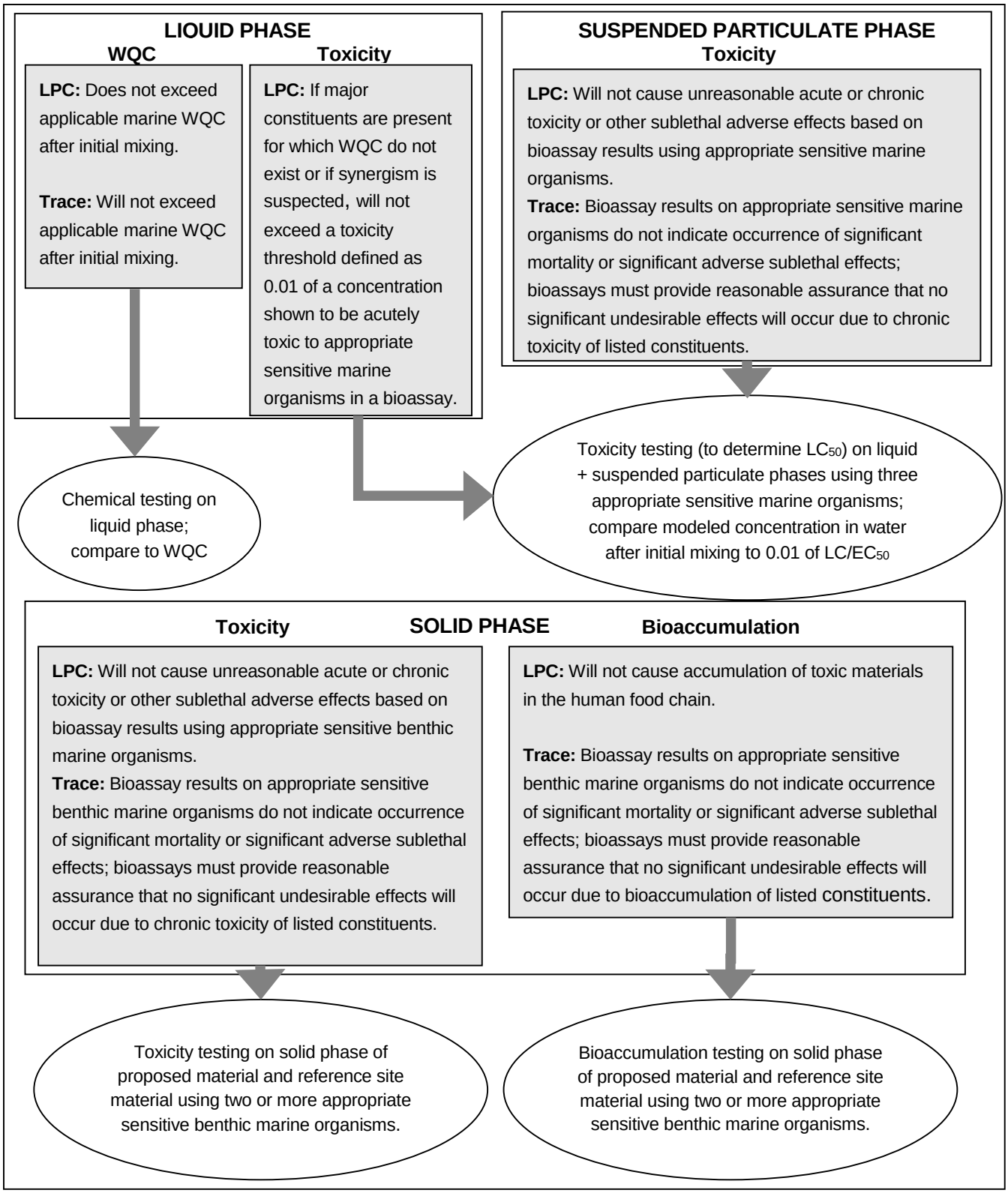
Solid: All material settling to the bottom of the above mixture within one hour.

## Box 2-1 Regulatory Evaluation of Dredged Material Proposed for Placement at HARS



## 3.0 ADMINISTRATIVE REQUIREMENTS

Applicants for Department of Army (DA) permits must provide the information necessary to process the permit and the information required to evaluate the impact of the proposed activity. The decision to grant or to deny a permit for disposal is based on a Public Interest Review of the probable impact of the proposed activity and its intended use. However, for permits authorizing discharge of dredged material into ocean waters, the applicant must demonstrate that the proposed disposal of dredged material will satisfy the environmental impact prohibitions, limits, and conditions set forth in the ocean-dumping regulations. The testing requirements set forth in this guidance document provide sufficient information to determine if the proposed discharge of dredged materials will meet or exceed the Limiting Permissible Concentration (LPC) (40 CFR 227.27).

The time limit for completing the work authorized by MPRSA dredged material disposal permits is three years from the date of permit issuance (33 CFR 325.6(c)). After the initial dredging event, a determination as to the acceptability of future dredged material placement at the HARS is required for any further dredging operation authorized by the permit. For most maintenance dredging projects, this revalidation does not automatically trigger a requirement for new sampling or testing of the sediment during the three year period. If a determination is made that no changed circumstances have occurred that warrant re-testing of the dredged material (e.g., chemical releases, oil spills, or other events that may have altered the character of the dredged material to the extent that the original test results may not be representative of the new material to be dredged) the previous characterization of the project material will be used to determine suitability of HARS placement without additional testing (e.g., see Section 2-2).

Prior to submitting the permit application and information required to process the permit, the applicant should call NYD personnel to discuss the testing program. Applicants need to request a sampling and testing plan from NYD prior to performing sampling and testing. Box 3-1 states the information needed for NYD to provide an applicant with a sampling and testing plan.

Application submissions and all other inquiries should be directed to:

United States Army Corps of Engineers
New York District, CENAN-OP-R
Regulatory Branch
26 Federal Plaza, Rm. 1937
New York, NY 10278-0090
(917) 790-8417

### 3.1 Requirements for Permit Applications

Applicants for DA permits for dredging and ocean discharge of dredged material must use the standard application forms:

For dredging sites located in New York State:

- Joint Application Form for Corps of Engineers and New York State, dated Feb 2013; and
- Environmental Questionnaire Form; and
- FCAF- Federal Consistency Assessment Form.

For dredging sites located in New Jersey:

- ENG Form 4345; and
- Environmental Questionnaire Form; and
- NJ Coastal Zone Form.

Application forms and instructions can be found on the NYD website at:

http://www.nan.usace.army.mil/Missions/Regulatory/ObtainingaPermit.aspx

In addition, all applicants must also submit the additional technical information described in this section and in Box 3-1. Application forms may be obtained from NYD. NYD personnel are also available for pre-application consultation.

For permits pertaining to dredging in navigable waters and placement at the HARS, the application must include: a description of the area to be dredged; type, composition, and quantity of the material to be dredged; the need for the dredging and analysis of non-ocean disposal alternatives; the method of dredging; the method of transportation and disposal; and a summary of past dredging and spills at the site. A summary of the technical information that must be submitted with an application for permits authorizing dredging and ocean disposal of dredged material is provided in Box 3-1.

The applicant is responsible for providing all information necessary to support the required evaluations. The remaining sections of this guidance document focus on the procedures for testing the proposed dredged material and on the supporting information and data that must be provided by the applicant.

**Box 3-1. Technical information (with supporting documentation) to be submitted with applications for DA permits for dredging and ocean discharge of dredged material**

- Site Plans of Area to be Dredged:
  - 8½" x 11" vicinity map showing area to be dredged in relation to overall larger geographic area and Harbor water bodies.
  - 8½" x 11" site-plan map with area to be dredged clearly marked.
  - 8½" x 11" cross-section or elevation view of the proposed dredging.
  - Photograph(s) of dredging site.
  - Four (4) paper copies of a hydrographic survey that is no more than six (6) months old. These surveys are to be provided on large scale engineering size drawings so that the underwater topography can be properly assessed, and the needed sediment sampling locations can be accurately plotted. The surveys need to include:
    - The location of outfall structures in the area to be dredged as well as into surrounding areas that would influence the area to be dredged, including a printed note with the volume and type of discharge.
    - The limits of the entire dredging area(s) footprint(s).
    - The proposed project dredging depths, dredging overdepth depths, and the total maximum dredging depth; all referenced to Mean Lower Low Water datum.
    - The different estimated volumes of bottom sediments to be dredged for transport to the ocean site; including the estimated required dredging volume, the estimated overdepth volume, and estimated total volume.
    - Indicate location of any wetlands, shellfish areas, or other special habitats.
    - Include latitude/longitude on survey map.
    - The date(s) that the hydrographic survey data was field collected and the date the survey was signed by the supervising engineer or licensed surveyor.
- Spill history:
  - An up-to-date history of spills (type, volume, date) in and nearby (within at least 1 mile) of the proposed dredging area(s) since last sampling date, available from the U.S. Coast Guard-Sector New York
  - HARS Placement Information Sheet (See Appendix C).
  - History of previous dredging (i.e., permits, volumes, disposal sites, dates).
- A discussion of purpose and need for the proposed dredging, including any benefits to be gained (or retained) by the proposed dredging.
- Alternatives Analysis:
  In accordance with 40 CFR Part 227.16(b), a well-documented discussion is required from the permit applicant that there are no practicable alternatives to ocean placement available to the applicant for placement of the proposed dredged materials. Ocean placement is considered the disposal method of last resort. The presumption in the regulation is that there is an available alternative.
- Method of Dredging, Transportation, and Disposal:
  - Type of dredging equipment (i.e. clamshell, hopper dredge).
  - Type of transportation equipment (i.e. split hull, pocket barge).
  - Capacity of the transport vessels.
- Type, Composition, and Quantity of the Dredged Material:
  - Volume of material to be dredged.
  - Types of dredging (i.e., maintenance, channel widening/deepening).
  - Composition of the dredged material (i.e. % sand, silt, and clay).

After joint review of the above information by NYD and EPA Region 2, NYD will provide a sampling and testing plan to the applicant. Applicants should not perform sampling without first obtaining a NYD approved sampling and testing plan.

## 3.2 Evaluation of Dredged Material Suitability

### 3.2.1 Sampling and Testing Requirements

Upon receipt of all of the technical information listed in Box 3-1, NYD will provide the applicant with a sampling and testing plan that has been coordinated with EPA Region 2. The sampling and testing plan will identify the specific locations from which samples must be taken, how the sediment samples are to be combined for testing, and a list of the physical, chemical, and biological analyses required to evaluate the sediment. If any changes are made to the dredging project (e.g., changed project depths or areas) prior to sampling, they must be coordinated with NYD so that corresponding changes may be made to the sampling plan to ensure that it continues to represent the material proposed for ocean placement.

An example of the checklist that is provided to applicants by NYD to communicate the specific testing that will be required for their project is provided in Box 3-2. Additional analytes or tests may be required on a case-by-case basis.

The methodologies required to sample sediment, conduct testing, and report data and testing conditions are described in Chapters 5-9 of this document. The applicant is responsible for providing all information necessary to support the required evaluations.

It is strongly recommended that, prior to contracting with any laboratory, applicants require that they provide written confirmation that the laboratory has reviewed the testing requirements laid out in this manual and that they can perform the required analyses to the specified control/acceptance criteria. Guidelines and checklists for conducting this demonstration of capability are provided in Appendices E and F.

**Box 3-2. Example of a checklist that is provided to applicants to communicate specific testing requirements**

Any box checked off indicates an analysis or assay that is required for a given project.
X = per homogenized project sediment core; HARS reference sediment composite; and control sediment composite(s)
C = per bioassay project sediment composite
W = Site water and elutriate
T = per tissue replicate (Reference, Test, Pre-test),

1. SEDIMENT PHYSICAL ANALYSIS
a. X **Grain Size Analysis** (% gravel, % sand, % silt, & % clay)
b. X **% Moisture**
c. C **Specific Gravity**
d. C **Bulk Density**
e. C **Plastic and Liquid limits (Atterberg limits)**

2. SEDIMENT CHEMICAL ANALYSIS
a. X **Total Organic Carbon** (%)
b. C **Metals:** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
c. C **PAHs (LMW):** (acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)
d. C **PAHs (HMW):** (benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)
e. C **Semi-volatiles:** (1,4 dichlorobenzene)
f. C **Pesticides:** (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I, II, and sulfate), heptachlor, heptachlor epoxide)
g. C **PCBs:** (congener nos. 8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, 209)
h. C **Dioxins/Furans:** (2,3,7,8 - substituted isomers, n=17)

3. CHEMICAL ANALYSIS OF SITE WATER AND ELUTRIATE
a. W **Metals:** (Ag, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
b. W **PCBs:** (congener nos. 8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, 209)
c. W **Pesticides:** (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans (I, II, and sulfate), heptachlor, heptachlor epoxide)

4. BIOASSAYS (species listed in guidance manual)
a. C **Water Column Acute Toxicity** (bivalve larvae*, A. bahia*, *Menidia* sp.)
b. C **10-Day Benthic Acute Toxicity** (*A. abdita*, *R. abronius*, *E. estuarius*, or *L. plumulosus*)
c. C **10-Day Benthic Acute Toxicity** (*A. bahia*)
d. C **28-Day Bioaccumulation** (*N. virens*, and *M. nasuta*)

**Box 3-2 cont. Example of a checklist that is provided to applicants to communicate specific testing requirements**

5. 28-DAY WHOLE-SEDIMENT BIOACCUMULATION TISSUE ANALYSIS
a. T **Metals:** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)
b. T **Pesticides:** (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I, II, and sulfate), heptachlor, heptachlor epoxide)
c. T **PCBs:** (congener nos.8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, 209)
d. T **Semi-volatiles:** (1,4 dichlorobenzene)
e. T **PAHs (LMW):** (acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)
f. T **PAHs (HMW):** (benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)
g. T **Dioxins/Furans:** (2,3,7,8 - substituted isomers, n=17)

### 3.2.2 Data and Report Submission

After testing has been completed, the applicant must provide NYD with a full report of sampling and testing performed in support of their application. This report is to be submitted in a suitable electronic format (e.g., pdf). Clear data summaries must be provided for all matrices, bioassays, and analyses. Data summaries must detail: all sample and test results, quality control sample results; pertinent sampling, extraction and analysis locations and dates; statistical analyses; supporting raw data[2]; and any other applicable calculated performance criteria as specified in this manual. Checklists which outline the minimum content requirements for final reporting of bioassay and chemical analytical data are included as Appendices F and G.

In addition, two hardcopies of an “executive summary” of the testing report must be submitted to NYD. The executive summary should include:

- Names of the laboratories and personnel that performed the tests;
- Charts and tables that clearly show locations from which samples were taken;
- Summary tables of physical, chemical, and bioassay results; including results for individual replicates and all control and reference treatments;
- Pertinent statistical comparisons and calculations;
- A discussion of any problems (and associated corrective actions) encountered, and departures from QA or protocols specified in this manual or a quality assurance project plan.

[2] Raw data are ". . . any laboratory worksheets, records, memoranda notes, or exact copies thereof, that are the result of original observations and activities or a study and are necessary for the reconstruction and evaluation of the report of that study." (40 CFR 792)

### 3.2.3 Evaluation of Test Results to Determine LPC Compliance

If the testing required by this manual shows that either the water-column or benthic LPC is not met for a proposed dredged material project, ocean placement of dredged material will not be approved. In such cases, the applicant must modify the proposed dredging and disposal activity (e.g., move the proposed work, realign proposed channels, alter project depths, change scow discharge volumes) and submit additional supporting data to be considered further under the existing permit request. Alternatively, the applicant may withdraw the proposed project from consideration for ocean placement and pursue a non-ocean alternative to manage the sediment.

#### 3.2.3.1 Water Column LPC Compliance (Liquid and Suspended Particulate Phase)

Specific criteria for water quality and water column toxicity must be met outside of the designated site at all times and for the entire water column after the 4-hour initial mixing period (40 CFR 227.29(a)). The mean concentration of any dredged-material constituent dissolved into the liquid phase of dredged material (estimated using the methods described in Chapter 9), after making an allowance for initial mixing, shall not exceed applicable marine water quality criteria. The concentration of dredged material remaining in suspension after discharge to receiving waters should not exceed 0.01 of the lowest concentration shown to affect 50% of organisms exposed in any bioassay test (i.e., 0.01 of the lowest $LC_{50}$ or $EC_{50}$).

Methods for preparing and evaluating the liquid phase and suspended particulate phase (SPP) are presented in Chapters 9 and 7, respectively. Dissolved concentrations of specific constituents are directly measured in the prepared liquid phase. Suspended phase toxicity is assessed by exposing three species of water column organisms (a fish, shrimp and bivalve larva) for 96 hours to a series of dilutions prepared by mixing dredged material with different proportions of dredging site and ocean water. The results of these analyses are used to calculate how much mixing will be required to meet the LPC for both water quality and toxicity.

A numerical model[3] that has been parameterized to simulate the water column mixing characteristics of the HARS is used by NYD and EPA to determine what operational limitations are to be placed (e.g., barge volume or boundary distance restrictions) to assure that LPC requirements are met following discharge. This model estimates water mixing, with respect to the SPP test data and information about the specific material and disposal equipment.

[3] STFATE is the model typically used to evaluate the initial mixing of a discharge into open water systems when disposal occurs as discrete discharges from barges and hopper dredges. A copy of this model and more information about this model can be obtained at http://el.erdc.usace.army.mil/dots/models.html.

#### 3.2.3.2 Benthic LPC Compliance (Solid Phase Toxicity and Bioaccumulation)

Solid phase toxicity and bioaccumulation potential are conducted by simultaneously exposing two groups of test organisms to sediments: one group of organisms is exposed to a representative sample of the dredged material that is proposed for ocean placement; the other group is exposed to samples of sediment collected from a reference area near the proposed placement site. Interpretation of benthic LPC compliance is conducted by comparing the magnitude of toxic effects and contaminant accumulation caused by the two exposures.

Reference sediment, used to test dredged materials proposed for placement at the HARS, is collected from a location south of the HARS (see Section 5.1.2). It is a natural sediment that represents conditions that would exist in the vicinity of the disposal site had no dredged-material disposal ever occurred, but having all other influences.

#### 3.2.3.3 Solid Phase Toxicity

Solid phase toxicity is assessed by exposing two species of crustaceans (an amphipod and a mysid shrimp) to the proposed dredged material, reference sediment and control sediment for ten days using the methods specified in Chapter 6.

Dredged material does not meet the LPC for benthic toxicity when 1) the mean mortality of any one species exposed to proposed dredged sediment exceeds the mean mortality observed with reference sediments by at least 10% (20% for amphipods) and 2) this difference is statistically significant (p=0.05).

#### 3.2.3.4 Solid Phase Bioaccumulation

Solid phase bioaccumulation is assessed by exposing two species of organisms (a worm and a clam) to the proposed dredged material, reference sediment and control sediment for 28 days using the methods specified in Chapter 8. The tissues of surviving organisms are then analyzed to determine the degree of contaminant uptake that resulted from exposure using methods specified in Chapter 9. (Due to the expense associated with running bioaccumulation tests, applicants may opt to start this testing after determining that the material meets the LPC regarding benthic toxicity).

If mean tissue concentrations for any contaminant of concern in organisms exposed to the dredged material are higher than mean concentrations in organisms exposed to the reference sediment and statistically significant at $\alpha=0.05$, then a review will be undertaken to determine the biological significance of that accumulation. Where appropriate, contaminant residues measured at the conclusion of the exposure period that are statistically greater than reference will be mathematically corrected prior to their use in assessing the material's suitability for ocean placement to account for the longer term exposures of benthic organisms expected following the materials' placement in the ocean (i.e., steady state).

## 4.0 QUALITY ASSURANCE

Quality Assurance (QA) is an integral component of any program or project collecting and/or using analytical data or information for environmental decisions. An effective QA program ensures that the test data are defensible and of sufficiently high quality to support the decisions being made.

### 4.1 Quality Assurance Program

The importance of a QA program to dredging studies is to ensure that collected data, required to make permitting decisions, is of known and documented quality. QA activities also ensure that quality control (QC) procedures have been implemented and documented. QA programs set standards for personnel qualifications, facilities, equipment, services, data generation, record keeping, and data-quality assessments. The function of this QA program is to ensure that contracted laboratories and samplers comply with procedures specified in the 1991 Green Book (see Chapter 14 in EPA and USACE 1991, and EPA 1987c) and this RTM. The QA oversight is the responsibility of the applicant, in coordination with EPA and NYD.

The applicant should ensure that their sampling contractor has a quality assurance program in place and that, at a minimum, provides for the following quality assurance considerations:

- Samples are collected in accordance with the Standard Operating Procedures (SOPs) and QA plan
- Sampling personnel are appropriately qualified and adequately trained and sufficient training records are maintained
- Sample locations and depths are properly documented to ensure representation of the dredging footprint described in the DA dredging permit application

Routine audits should be conducted by the contractor's QA officer or delegate to ensure that all aspects of the sampling accurately reflect the work that was planned and completed, and that all necessary information, as defined by regulations, SOPs, or program-specific QA plans, is included. Results of audits should become part of the contractor's project file.

Laboratories are responsible for the analytical data they generate/report. All laboratories performing analyses should have established and should maintain a quality system. A quality system is the means by which an organization manages its quality aspects in a systematic, organized manner and provides a framework for planning, implementing, and assessing work performed by an organization and for carrying out required quality assurance and quality control activities.

Analytical data packages must have a signature of the laboratory manager, quality assurance officer or designated analyst verifying that the analytical data provided are valid. The applicant should ensure that their contracted testing laboratory, at a minimum, provides for the following quality assurance considerations:

- Tests are performed in accordance with the SOPs and QA plan
- Laboratory personnel are appropriately qualified and adequately trained and that sufficient training records are maintained
- Data are verified to ensure traceability between raw and reported data

Routine audits should be conducted by the laboratory's quality assurance unit to ensure that all aspects of the testing accurately reflect the work that was planned and completed, and that all necessary information, as defined by regulations, SOPs, or program-specific plans, is included. Results of audits should become part of the testing laboratory's project file.

### 4.2 Quality Assurance Plan Development

Pursuant to Order CIO 2105.0, May 5, 2000, "…it is EPA Policy that all environmental programs performed by EPA or directly for EPA through EPA funded extramural agreements shall be supported by individual quality systems…." Though not funded by EPA, this program requires EPA Region 2 to use data collected directly from applicants for the determination of permitting the dredged material to be placed at the HARS. Due to this critical use of the data and the potential for the dredged material to have a direct impact to the environment, EPA recommends that the contractor prepare a Quality Assurance Plan (QA Plan) to define the proper quality controls for their operations. A copy of the final QA plan should be submitted to EPA Region 2 to be retained in the QA Program files.

The QA Plan should be developed in accordance with *EPA Guidance for Quality Assurance Project Plans (QA/G-5)* (EPA 2002) and should be signed by an authorized representative of the organization performing the work.

A QA Plan should be prepared for the sampling components as well as the laboratory components of the project. If the organization performing the sampling is the same as the organization performing the analysis one QA plan may be prepared to cover both the sampling and laboratory analysis.

A QA Plan should clearly define the tests that will be conducted, who will conduct the work, and how results of the work will be reported. The QA Plan should be detailed enough to fully identify all roles and responsibilities, quality-assurance (QA) objectives, and how the QA objectives are to be achieved. The plan should be written so that a technical person unfamiliar with dredged-material evaluations can understand the objectives of the work and how the objectives will be met. The QA Plan assigns responsibility and ensures that all participants possess a thorough understanding of the work. The QA Plan should be composed of standardized elements covering the entire project from planning, through implementation, to assessment. The EPA QA/G-5 describes the specific elements to be included. The QA Plan should include but not be limited to:

- Clearly defined QA objectives that are consistent with the regulations, the Green Book, this regional guidance manual and the permit application;
- Descriptions of all technical procedures for field sampling, laboratory analyses (e.g.,

biological, chemical, physical), data reduction and validation, and reporting;
- Clearly written standard operating procedures (SOP) for all field and analytical procedures;
- Mechanisms for conducting performance and systems audits during the course of the field and laboratory work;
- Procedures for detecting problems with the sampling and analytical work, and implementing corrective actions in a timely manner.
- Procedures to demonstrate success of compliance with corrective actions

Guidance documents for the development of the QA Plan are provided at the following URL: http://www.epa.gov/QUALITY

While most QA Plans will describe project-specific activities, there are occasions when a *generic* QA Plan is more appropriate, such is the case with this program. A generic QA Plan addresses the general, common activities of a program that are to be conducted at multiple locations or over a long period of time; for example, it may be useful for monitoring programs that use the same methodology at different locations. The generic QA Plan describes, in a single document, the information that is not site-specific or time-specific but applies throughout the program. Site- and time-specific information for each individual project will be documented in the Sampling Scheme and List of Required Testing that is prepared by NYD and Region 2.

An organization that wishes to routinely perform work and submit data to NYD and Region 2 may prepare a generic QA Plan and provide a copy of the plan to NYD and Region 2. A generic QA Plan should be reviewed by the preparing organization annually to ensure that its content continues to be valid and applicable to the program. If changes are to be made to the generic plan, the revised generic QA plan along with an itemized list of the changes should be distributed to NYD, EPA and all parties involved with the sampling and testing.

### 4.3 Quality Assurance Plan Review Process

The applicant is responsible for their contractors (laboratories and/or sampling consultants) adhering to all applicable sections of the QA Plan. NYD reserves the right to conduct its own QA evaluations and data audits during the sampling and testing activities, as it deems necessary. The contracted organization is responsible for making the necessary provisions to allow for NYD inspections of field-sampling and laboratory activities. Any deviations from this guidance manual and QA Plan should be documented and coordinated with NYD prior to initiation of sampling or if work is already in progress, prior to the continuation of sampling or analyses.

## 5.0 FIELD SAMPLING AND SAMPLE HANDLING

All methods and procedures to be used in the field and laboratory with respect to sample collection, handling, and preservation should be outlined in the QA Plan and SOPs. Written protocols for the following steps may be referenced in the QA Plan:

- Preparation and use of sampling equipment and facilities
- Sample collection (sediment, water)
- Sample homogenization
- Sample compositing
- Sample preparation and manipulation
- Sample labeling and custody, transport, storage and disposal
- Sample preservation methods
- Sample holding times (including before and after extraction)
- Field sampling log entries
- Deviations from standard methodologies

Table 5-1 shows the various sources from which water and sediment will have to be collected to conduct the testing described in this manual.

**Table 5-1. Sediment and Water Samples Required to Conduct Testing Described in this Manual**

| Tests | Water Samples | | | Sediment Samples | | |
|---|---|---|---|---|---|---|
| | Disposal Site | Dredging Site | Control[a] | Dredging Site | Ref. Site | Control[a] |
| Water column | ● [b,c] | ● | ● | ● | | |
| Benthic | | | | ● | ● | ● |

[a] May or may not be field-collected
[b] Determine WQC compliance
[c] Dilution water; artificial or clean seawater may also be used. Laboratory must show that the artificial seawater is not toxic to water-column test organisms

## 5.1 Sediment – Sampling and Handling

### 5.1.1 Dredged Material

Because contaminant levels may vary across different depths of a sediment shoal, coring methods are required to representatively sample dredged material. The choice of appropriate equipment for use in core sampling a specific project depends upon several factors including: sediment type; required penetration; water depth; and the currents present at the project site. Cores should be collected using an appropriate core liner.

The sampling plan will specify the number (at least three) and location of core samples required for a given project area and the required depth to which cores must be taken. Core sampling to 2 feet below project depth will generally be required to allow for authorized overdepth dredging for proposed dredging projects. If collection of a single core sample from each location contributing to a composite yields insufficient sediment volume to conduct the tests required by this manual, multiple core samples can be taken from each location. However, the composite must be comprised of the same number of cores from each location. Failure to comply with the sampling plan will invalidate test results.

It is very important that sufficient sample material be collected to conduct all of the required tests, and that the collection and handling operations do not contaminate the samples. If difficulties obtaining samples from the specified locations are encountered in the field, or it appears that the NYD provided sampling plan will provide an insufficient volume of sediment to meet testing requirements, the applicant or contractor should notify NYD immediately for instructions (e.g., additional or alternative sampling stations).

#### 5.1.1.1 Individual Core Sampling and Handling

All individual core samples of the dredged material must be visually inspected prior to extruding the sediment from the core liner for sub-sampling, homogenization or compositing. Observations regarding the color, texture, odor, apparent stratification, and the presence of oil sheens or other contaminants (e.g., metal shavings, debris) should be recorded in the field notebook. NYD requires that contractors also take photographs of representative cores from each sampling location. Photographs must clearly document the sediments. Photos must be taken so that no shadows are present across the exposed core surface. Photographs must be taken directly over the cores; oblique angle photographs are not allowed. The image quality should be sufficient to easily identify basic sediment types within the core. Any discernible sediment strata greater than 2 feet in depth may be required to be sampled and analyzed separately. If the sampling scheme does not indicate a need to stratify within a core[4] but there are obvious demarcations, such as a silty upper layer and a sandy bottom layer, the applicant and/or the contract laboratory must contact NYD immediately to

[4] In some cases, state agencies may require the bottom half foot of the core to be analyzed in support of their water quality certificate.

determine if the strata should be analyzed separately.

Following visual inspection, the sediment in the core liner should then be extruded into appropriately sized and clean containers (e.g., HDPE buckets lined with food grade or other chemically clean liners) for transportation to the laboratory. Individual core samples (or stratum) should then be thoroughly homogenized and samples taken for physical parameters (see Section 9.1). The presence of any indigenous organisms, debris, or rocks (>1.0 mm) should be noted in the field log and then carefully removed from the sample. An archived subsample representative of each core (or stratum) should be kept in case additional or repeat analyses are warranted.

#### 5.1.1.2 Compositing of Core Samples

Core (or strata) samples are generally required to be combined together into composite samples for biological and chemical testing. As stated above, the composite must be comprised of the same number of cores from each core sampling location. If cores are sub-sampled, the contributions from each core to the composite should be proportional to the core length until sufficient composite volume is prepared.

### 5.1.2 Reference Sediment

Reference sediment must be collected at the Mud Dump Site Reference Site, located at 40º 20' 13"N, 73º 52' 11"W (40° 20.217´N, 73° 52.183´W). The reference site is located approximately 1.2 miles south of the HARS, in about 70 ft of water. Surface sediment grab samplers can be used to collect reference sediment. Sampling devices should be acid-rinsed (10% nitric acid) and solvent-rinsed prior to use. NYD recommends that Teflon®-coated samplers be used. Alternatively, reference sediments should be removed from the center of the grab to eliminate sediment that has come into contact with the sampling device.

The reference sediment is predominantly sand (>95%) with a minor silt and clay component (<5%), low moisture content (~23%) and negligible TOC (<0.2%) (SAIC,1993). Very fine (4-3 phi) and fine grains (3-2 phi) dominate (>68%) the sand fraction. If black mud, silt, coarse-grained sand or sand interspersed with silt lenses are included with the sample, it is not representative of Mud Dump Reference Site sediment and should not be used for testing purposes. Using inappropriate reference sediment invalidates testing.

### 5.1.3 Control Sediment

The purpose of control sediment is to confirm the biological acceptability of test conditions and to help verify the health of the organisms during the test. Therefore it is imperative that sediment collected for use as control sediment have no discernible negative influence on the test organism. Control sediment must be essentially free of contaminants and compatible with the biological needs of the test organisms (e.g., appropriate grain size). Sediment collected from sites where field-collected test organisms were obtained or sediment that the organisms were shipped or cultured in

may serve as appropriate sources of control sediment (for biological testing, pre-test sieving at the testing laboratory is necessary to remove indigenous organisms).

### 5.1.4 Storage, Handling, and Preservation of Sediment Samples

Sediments collected to support HARS evaluations will have to be sub-sampled to provide aliquots for all of the analyses required. The various sediments collected, however, are not subjected to the same suite of analyses. Table 5-2 presents the analyses typically required of each sediment type.

**Table 5-2. Analyses Typically Required for Sediment Samples Used for HARS Evaluation[1]**

| | Core (or stratum) | Composite Dredged Material | Reference Sediment | Control Sediment(s) |
|---|---|---|---|---|
| Physical Analyses[2] | | | | |
| Grain size | ● | ● | ● | ● |
| % Solids/moisture | ● | ● | ● | ● |
| Specific gravity | | ● | | |
| Bulk density | | ● | | |
| Atterberg limits | | ● | | |
| Bioassays | | | | |
| Suspended particulate phase toxicity | | ● | | |
| Solid phase toxicity | | ● | ● | ● |
| Bioaccumulation | | ● | ● | ● |
| Chemical Analyses | | | | |
| TOC | ● | ● | ● | ● |
| Organic contaminants | | ● | | |
| Inorganic contaminants | | ● | | |

[1] In some cases, NYD may require additional analyses not listed in table or require that analyses be conducted on core samples, or reference and control sediments
[2] Physical analyses are to be conducted prior to press sieving the sample.

Field-collected sediment must be sieved using a 2.0-mm (or smaller) mesh size sieve to effectively remove indigenous organisms, debris, and rocks. Subsamples of the sediment that are to be used in amphipod and mysid solid phase testing (see Section 6.4) may require additional sieving using a 0.5 mm sieve if other organisms, including potential predators or indigenous amphipods, are visible in the sample. The sample should then be sub-sampled into appropriate containers (see Tables 5-3- and 5-4) to provide laboratories with aliquots for all required analyses. Sediment should be tightly capped and stored at 4 $^{o}$C until ready for use.

Tables 5-3 and 5-4 list sample container, preservation method, and holding time requirements for

sediment samples to be used for biological, physical and chemical analyses (including for preparation of SPP and chemical elutriate). Specific provisions are necessary for the various required chemical analyses and analyte classes. Laboratories may propose alternate sample containers, preservation methods and/or holding times. However, these proposals should be based on an established reference such as an EPA Method, ASTM Method, or Standard Methods. The complete reference citation or a detailed description of the proposed alternate method must be included in the QA Plan for EPA review prior to commencement of the project.

**Table 5-3. Storage and Preservation of Sediment Sample Aliquots for Physical Analysis and Use in Bioassay Testing**

| Analysis | Volume Required/ Sample Container | Sample Preservation | Maximum Holding Time |
|---|---|---|---|
| Bioassay Testing | 170-L; HDPE buckets | Cool, 4 °C[1] | 8 Weeks[2] |
| Physical Parameters | 2-L; No specific container requirement | Cool, 4 °C[1] | 1 Year |
| Preparation of SPP/ chemical elutriate | 4-L, plastic | Cool, 4 °C | 8 Weeks[3,4] |

[1] Sediments may not be frozen prior to bioassay or physical testing.
[2] Sediments should be used as soon as possible to obviate the need for resampling should a test need to be repeated. The holding time for a composite sample begins with the collection date/time of the sample used in the composite that was collected the earliest. Once SPP bioassay elutriate is prepared, the biological tests must be started within 24 hours.
[3]SPP must also be prepared within 14 days of site water collection.
[4]Chemical elutriate should be prepared within eight weeks of sediment collection, however if an elutriate test must be repeated at a later date due to QA issues, sediment that has been kept frozen in the dark at ≤-20 °C for less than one year can be used to prepare it. In all cases, chemical elutriate must be prepared using water collected within 7 days.

**Table 5-4. Storage and Preservation of Sediment Samples for Chemical Analysis**

| Analyte | Sample Container | Sample Preservation[1] | Maximum Holding Time[1,2] |
|---|---|---|---|
| Organic Compounds ( Pesticides, PCBs, PAHs, Dioxins, Furans) | 4 oz. wide mouth amber glass with Teflon® lined lid (fill 75%) | 1) Cool, 4 °C Or | 1) 14 days to extraction Or |
| | | 2) ≤ -20 °C; Keep in dark | 2) 1 Year |
| Total Organic Carbon (TOC) | 4 oz. wide mouth amber glass or clear glass wrapped in foil (fill 75%) | 1) Cool, 4 °C Or | 1) 28 days to extraction Or |
| | | 2) ≤ -20 °C; Keep in dark | 2) 1 Year |
| Total Metals | 4 oz. wide mouth glass (amber optional) or plastic (fill 75%) | 1) Cool, 4 °C Or | 1) 6 months Hg - 28 days Or |
| | | 2) ≤ -20 °C Keep in dark optional | 2) 1 Year |

[1] Sediment sample must be stored and maintained in an environment (i.e., in a cooler with ice or refrigerator) at 4 ± 2 °C, immediately upon generation/collection. Alternatively, sediments for chemical analysis (or preparation of chemical elutriate in some cases) may be frozen and maintained in a storage condition at -20 °C for up to one year.
[2] The holding time for a composite sample begins with the collection date/time of the sample used in the composite that was collected the earliest.

## 5.2 Water – Sampling and Handling

### 5.2.1 Sampling Devices and Methods

Water samples taken for use in testing should be collected with either a non-contaminating, oil-free pump or a discrete water sampler. When sampling with a pump, the potential for contamination can be minimized by using a peristaltic or a magnetically coupled impeller-design pump. The system should be flushed with the equivalent of 10 times the volume of the collection tubing prior to collection of the sample. Also, any components within several meters of the sample intake should be non-contaminating (i.e., Teflon®, or sheathed in polypropylene or epoxy-coated). Potential sample contamination from the survey vessel (e.g., emissions and water discharge) and any apparatus used in sampling should be avoided. Discrete water samplers should be of the close/open/close type so that only the target water sample comes into contact with internal sampler surfaces. Seals on the

water samplers should be Teflon®-coated whenever possible. Discrete water sampling devices should be acid-rinsed (10% nitric acid) and solvent-rinsed prior to use. Samples should be collected into bioassay-grade Cubitainers® or appropriately sized HDPE buckets lined with food-grade or other chemically clean liners. Samples may be shipped to laboratories in Cubitainers® for eventual transfer into sampling containers listed in Table 5-5.

### 5.2.2 Dredging Site Water

Water obtained from the dredging site collected from each sampling and testing unit identified in the plan provided by NYD will be mixed with dredged sediment from that unit (see Sections 7.4 and 9.2.2) to prepare the SPP used in bioassay testing and to prepare an elutriate sample for chemical analysis of dissolved contaminants of concern in the liquid phase. This subsurface water sample must be collected from within the boundaries of the dredging site and within one meter of the surface. Water samples should be taken at high slack tide and should avoid periods during which sediment resuspension due to coring activities may introduce project sediment into the sample.

### 5.2.3 Control/Overlying/Dilution Water

The function of control water as a water-column bioassay control treatment is analogous to control sediment as a benthic control treatment. This water is used as the overlying or dilution water in bioassay testing, including control exposures. Control water should be the same water in which the test organisms are held prior to testing. Testing laboratories are responsible for collecting appropriate control water from the field or creating it from artificial sea salt mixtures. Control water must be analyzed annually for the parameters specified in ASTM 2002a.

Laboratories must also construct and maintain control data files from the results of the control treatments. If water is collected in the field, it should be collected prior to sediment collection (if applicable) at one meter below the surface. If tank trucks are used to haul water to the testing laboratory, a certification of tank cleanliness (dedicated for clean saltwater or appropriately decontaminated food grade tanks only) should be provided for each shipment. The salinity of each batch of saltwater must be checked before using it for testing and adjusted using sea salts if it is too low or diluted with laboratory grade, analyte-free, deionized water if the salinity is too high.

### 5.2.4 Storage, Handling, and Preservation of Water Samples

Site water and dilution water samples must immediately be stored in appropriate, certified chemical-free containers and maintained in an environment (i.e., in a cooler with ice or refrigerator) at 4 ± 2 °C until they are analyzed or used to make the chemical elutriate and SPP (or until dilution water is used in the solid phase bioassays). SPP must be prepared within 14 days, and chemical elutriates must be prepared within seven days of collection of site water.

SPP and chemical elutriates are prepared using site water and the composite sediment sample of dredged material by combining one part sediment with four parts water (volume/volume) and

vigorously mixing it for 30 minutes. Ideally, the SPP and elutriate would be obtained from the same batch of supernatant. To accomplish this, sufficient water and sediment would be mixed and allowed to settle for an hour. After settling, a portion of the supernatant is removed to serve as the SPP and the remaining supernatant is filtered through a 0.45 micron mesh filter to serve as the chemical elutriate. More detailed instructions for preparing the SPP and elutriate are presented in Sections 7.4 and 9.2.2, respectively, of this manual.

Table 5-5 lists the laboratory sample container, preservation and holding time requirements for water (and elutriate) samples to be used for chemical analyses. As stated in Section 5.2.1, water should be collected using appropriate containers and shipped to laboratories in Cubitainers® for eventual transfer into the listed sampling containers. Table 5-6 lists the sample container, preservation and holding time requirements for water samples to be used for SPP preparation. Note that requirements differ for specific analyte classes. Laboratories can propose to employ alternate sample containers, preservation and/or holding times. However, these should be based on an established reference such as an EPA Method, ASTM Method, or Standard Methods. The complete reference citation or a detailed description of the proposed alternate method must be reviewed and accepted by EPA prior to commencement of the project.

**Table 5-5. Storage and Preservation of Site Water and Elutriate Samples for Chemical Analysis**

| Analyte | Sample Matrix | Volume Required/ Sample Container[2] | Sample Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Organic Compounds (Pesticides, PCBs) | Site water & elutriate[1] | 1 L amber glass | Cool, 4°C | 7 days to extraction |
| Total Metals | Site water & elutriate[1] | 250 mL rigid plastic or glass | $HNO_3$ to pH <2 | 6 months |
| Mercury<br>or | Site water & elutriate[1] | 250 mL rigid plastic or glass | $HNO_3$ to pH <2 | 28 days |
| (Method 1631e) | Site water & elutriate[1] | 125 mL Teflon | HCl to pH <2 | 90 days |

[1]Elutriate must be prepared within seven days of water sample collection for organic compounds and metals. Also see Tables 5-3 and 5-4 for relevant sediment holding times.
[2]Site water and elutriate samples must be analyzed in triplicate therefore three of each bottle type is necessary.

**Table 5-6. Storage and Preservation of Water Samples for SPP Preparation**

| Test | Sample Matrix | Volume Required/ Sample Container | Sample Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Suspended Particulate Phase | Site water[1] | 10L – HDPE (fish)<br>10L – HDPE (invertebrate)<br>2L – HDPE (zooplankton) | Cool, 4 °C | 14 days[2] |

[1]Site water used to prepare 100% SPP for all tests and diluted with laboratory control/dilution water

[2]Prepared SPP must be used in tests within 24 hours of preparation.

## 6.0 BENTHIC (SOLID PHASE) TOXICITY TESTING

All methods and procedures to be used in assessing the solid phase toxicity of dredged material proposed for placement at the HARS should be detailed in the QA Plan. Written protocols should be referenced to assure that data quality objectives are met for each of the following test conditions:

- Consistent sensitivity (reference toxicant effects) and health (control survival) of test organisms;
- Acceptable water quality (temperature, salinity, pH, dissolved oxygen, ammonia, flow rate) as specified in test protocols; and
- Appropriate number of replicates and frequency of observations as specified in the test protocols.

### 6.1 Introduction

Solid phase toxicity tests are designed to determine whether the proposed dredged material is likely to produce unacceptable adverse effects on the benthic marine environment. In the acute solid phase toxicity tests, one species of amphipod and one species of mysid shrimp are exposed to the proposed dredged material, reference sediment, and laboratory control sediment for ten days. After 10 days, the number of survivors in each exposure chamber are recorded and statistically compared.

*Modifications to any of the procedures and test conditions outlined in this section must be approved in writing by EPA Region 2 before they are incorporated as standard operating procedures.*

### 6.2 Species Selection

Two test types are required: A test using one of four amphipod species (either *Ampelisca abdita*, *Rhepoxynius abronius*, *Eohaustaurius estuarius*, or *Leptocheirus plumulosus*), and the other using the mysid, *Americamysis bahia* (Table 6-1). *A. abdita* is the benchmark amphipod test species for all NYD projects and must be used whenever possible. If *A. abdita* is not available then approval must be secured in writing from NYD for use of one of the alternate amphipod species from those listed in Table 6-1. Details of suppliers contacted, date contacted and the reason for unavailability of *A. abdita* must be provided to support the request.

Each lot of organisms obtained from a commercial supplier must be taxonomically verified. Laboratory reared organisms from the testing laboratory must be verified on an annual basis. Each batch of organisms must be positively identified. A citation of the taxonomic key and the distinguishing characteristics used during verification must be documented.

The testing laboratory should have established a reference toxicity testing record for any species selected for use in dredged material testing (a minimum of five reference toxicant tests with results within laboratory acceptability limits of ± 2 SD and five control samples with acceptable survival and/or development).

**Table 6-1. Test Species for Solid Phase Toxicity Tests**

| | |
|---|---|
| Amphipods (Choose one) | |
| *A. abdita** | Subadults, 3-5 mm, retained on 0.7 mm sieve but passing through a 1.0 mm sieve. |
| *R. abronius* | 3-5 mm, retained on 1.0 mm sieve with large individuals (≥ 5 mm) excluded. |
| *E. estuarius* | 3-5 mm, retained on 1.0 mm sieve with large individuals (≥ 5 mm) excluded. |
| *L. plumulosus* | Subadults, 2-4 mm, retained on a 0.5 mm sieve but passing through 0.7 mm sieve. |
| Shrimp | |
| *A. bahia** | 1-5 days old; age difference within batch to be 24 h or less |

*Preferred Species. *L. plumulosus* is the alternate amphipod species when test sediment is composed of more than 5 % mud (silt + clay) and no greater than 85% clay. *E. estuarius* and *R. abronius* are possible alternates when the mud content (silt + clay) of the test sediment is less than 5 %.

### 6.3 Organism Handling/Acclimation

a) Organisms should be held a minimum of 48 hours prior to bioassay testing.

b) Organisms should be acclimated to test conditions gradually. Temperature should not change by more than 3 $^{o}$C and salinity should not change by more than 3 ‰ over any 24 hour period.

c) Organisms should be held, for at least 24 hours before using them in tests, in sieved collection site sediment or other non-toxic sediment and overlying water at the required temperature and salinity that will be used in the test.

d) Overlying water should be renewed every 24-48 hours during acclimation.

e) Temperature, pH, salinity, and dissolved oxygen must be measured daily. Water quality parameters should be recorded before and after renewal of holding water.

f) It is recommended that amphipods be fed daily during acclimation (amphipods are not fed during the test). *Phaeodactylum tricornutum* (approximately 2.5 x $10^5$ cells/mL) or *Isochrysis* sp. (concentrated) have been used successfully to sustain *A. abdita* during holding and acclimation, although any food source, including commercially prepared sources, with which a testing laboratory has documented success is acceptable.

a) Mysids must be fed daily (approximately 100 *Artemia* nauplii per mysid) during acclimation as

a precaution against cannibalism. Mysids should not be overfed as this could cause unacceptable water quality conditions.

b) If mortality exceeds 10 percent over the entire acclimation and holding period, or 5 percent over the final 24 hours of acclimation, then it is recommended that the lot of organisms be discarded and the acclimation be started with a new batch of organisms.

## 6.4 Sediment and Water: Handling and Preparation for Solid Phase Tests

### 6.4.1 Overlying Water

Test chamber overlying water should be the same water in which the test organisms are held prior to testing. Testing laboratories are responsible for collecting suitable water (see Section 5.2.3) from the field or preparing it using artificial sea salt formulations. If artificial sea salts are used, they must not contain EDTA or sodium thiosulfate, as they could affect contaminant availability in the test sample matrix. All seawater must be aerated (if stored in large quantities) at the laboratory and/or kept at 4 $^{o}$C. Records must be maintained to document methods used to collect and/or prepare control water and conditions under which control water is kept in the laboratory.

### 6.4.2 Control Sediment

The purpose of the control sediment is to confirm the biological acceptability of the test conditions/procedures and to help verify the health of the organisms during the test. Therefore, it is imperative that the control sediment have no discernible negative influence on the test organism.

Each testing laboratory must provide the appropriate control sediment(s). The sediment can be collected from uncontaminated sites where field-collected organisms were obtained. If the organisms are laboratory reared or purchased from a supplier, then the control can be the sediment in which organisms were shipped or cultured. Field-collected control sediments must be press sieved through a 2.0 mm or smaller mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in solid phase testing. Control sediments for *Ampelisca* spp. testing must be press sieved through a 0.5 mm sieve prior to use. Control sediment handling procedures must be documented in the final testing report.

### 6.4.3 Reference Sediment

Reference sediment should be press sieved through a 2.0 mm or smaller mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in solid phase testing. Reference sediments for *Ampelisca* spp. testing must be press sieved through a 1.0 mm sieve prior to use. A final sorting of the sieved reference sediment over a light table is recommended. Reference sediment handling procedures must be documented and included in the raw data of the final report.

Reference sediment must not be held longer than eight weeks before it is used in testing.

### 6.4.4 Test Sediment (Dredged Material)

The dredged material sample should be press sieved using a 2.0 mm or smaller mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in solid phase testing. Test sediments for *Ampelisca* spp. testing must be press sieved through a 0.5 mm sieve prior to use if live organisms (e.g., other amphipods or predatory worms) are noted in the composite. Homogenize the sediment until a consistent sediment color and texture are obtained. The sample should be tightly capped following sieving and homogenization and stored at 4°C until ready for use.

Sediments for biological evaluation must be tested within 8 weeks of collection. Sample holding time starts as soon as the first sediment core contributing to a composite is collected. Test sediments should be used in biological testing as soon as possible to avoid the need for resampling in the event bioassays need to be repeated.

### 6.4.5 Ammonia Purging Procedures

In laboratory sediment toxicity tests, ammonia may be present at, or increase to levels in sediments or overlying water that are toxic to test organisms. Ammonia is not a solid phase contaminant of concern in dredged material disposed of in open water[5], but its potential to cause toxicity during the test can confound the ability to confidently attribute any observed toxicity to persistent sediment contaminants in the dredged material. This must be mitigated by adopting procedures to ensure that ammonia concentrations are below toxic thresholds prior to initiating exposures and that they remain below potentially toxic concentrations throughout the exposure period.

For most test species, the un-ionized form of ammonia is the more toxic form. However, given the expected range of pH, test temperatures, and salinity, initial management of ammonia based on total ammonia pore water concentration is expected to be sufficient to effectively manage potential interferences in solid phase tests before exposures are initiated. Once initial pore water total ammonia is reduced to <20 ppm in the test sediment, toxic levels of ammonia are not expected to be present in the pore water or overlying water during the 10-day exposures, even if the test chambers are not renewed (Ferretti *et. al*. 2000; Ferretti 1999).

If pre-test ammonia concentrations exceed toxic thresholds, purging procedures are performed **prior** to setting up the test chambers. Reference and control sediment(s) must be treated similarly to test sediment to mimic the ammonia purging process. Reference and control sediment pore-water total ammonia do not need to be measured unless desired, but the overlying water must be renewed at the same frequency and handled similarly to the test sediment. The recommended method for reducing ammonia levels prior to testing is described in the remainder of this subsection. A major advantage of this method is that it simultaneously reduces ammonia for both test species. Any

[5] Despite being highly toxic to marine organisms, ammonia is not considered a contaminant of concern in the solid phase of dredged material because it is non-persistent and does not bioaccumulate following discharge at open water sites.

alternative method used must have a demonstrated ability to effectively reduce ammonia concentrations, while not impairing sediment contaminant availability.

Three liters (by volume) of sieved test composite sediment should be spread evenly to a depth of 7-11 mm over the bottom of a 20" x 17" x 5" high-density polyethylene (HDPE) plastic pan. Different size trays/pans can be used for purging; however, a 7-11 mm sediment depth must be maintained and overlying saltwater must be added to produce a ratio of 3 parts water to 1 part sediment. Place a thin plastic cover (Clear Sheet Lexan® Polycarbonate or Vivak®, 0.04 - 0.06 inches thick or equivalent) over the surface of the sediment to reduce turbulence and carefully add 9.0 L of clean seawater. The same source of water that will be used for overlying water in the 10-day test(s) should be used for ammonia purging. Carefully remove the plastic turbulence reducer. Cover the top of the HDPE basin and aerate the overlying seawater. It is possible that more than one tray may need to be prepared to ensure sufficient sediment volume for the 10-day tests and ammonia monitoring surrogate chambers. Surrogate chambers are treated the same as the test chambers throughout the test but no organisms are added.

Overlying water should be renewed, and pore water total ammonia concentrations measured, every 24 hours by gently decanting or siphoning overlying water from the low side of the purging tray after propping up opposite end of purging basin. Remove 100 mL of sediment (or other appropriate amount) with a spatula for centrifugation and smooth the remaining sediment surface and refill the ammonia purging basin with saltwater as described in preceding paragraph.

Once pore water total ammonia is measured to be less than 20 ppm in the dredged material composite, two additional representative subsamples of the sediment should be analyzed to confirm the initial measurement. If confirmed, then sediments can be distributed to test and surrogate chambers. If the average pore water total ammonia of the three analyses is >20 ppm, carefully refill the purging basins/trays with saltwater using the plastic turbulence reducers and continue the ammonia purging process recording ammonia, salinity, dissolved oxygen, temperature, and pH levels, and renewal dates and times.

Overlying water renewals during purging is limited to once daily. For most dredged material composites, the purging procedures described above should reduce total pore water ammonia concentrations to <20 ppm in 1 to 7 days. Data from the entire purging process must be recorded.

### 6.5 Solid Phase Toxicity Test Procedures

Initial pore water total ammonia concentrations must be measured in test composite sediments prior to setting up and initiating the solid phase toxicity tests. Total ammonia concentrations must be <20 ppm before the 10-day tests can be initiated (See preceding section).

#### 6.5.1 Test Setup

Each series must include a minimum of five replicates of test sediment, five replicates of reference

sediment, and five replicates of control sediment. A minimum of 20 organisms per replicate are required.

All sediment samples should be completely mixed immediately prior to introduction to test chambers.

After sediments are placed in test jars, overlying water should be added using a plastic disk or other procedure to minimize disturbance to the sediment. Each chamber should be loosely covered and aerated (approx. 100 bubbles/min). The test chambers should be allowed to settle overnight before adding test organisms.

A set of eleven 'surrogate' chambers (i.e., test sediment and water with no stocked organisms) for each dredged material sample tested must also be included in the test design to allow for once a day monitoring of overlying and pore water ammonia during the test (even if initial total ammonia is <20 ppm in the pore water). If mysid and amphipod tests are conducted concurrently, a single set of surrogate jars per test sediment will suffice (overlying water for mysids and pore water for amphipods). Surrogate chambers for ammonia monitoring are not required for reference and control samples.

### 6.5.2 Test Conditions

All solid phase toxicity tests are conducted as static non-renewal exposures. All testing chambers (including surrogate, reference, and control chambers) should be treated exactly the same throughout the course of the 10-day test. All chambers must be aerated (approx. 100 bubbles per minute) during the 10-day organism exposure. Daily records must be maintained for salinity, temperature, DO, pH and obvious mortalities. Formation of tubes or burrows (amphipods), amphipod emergence from sediment, and any physical or behavioral abnormalities must also be recorded.

A summary of test conditions are provided in Tables 6-3 and 6-4 for mysids and amphipods, respectively.

#### 6.5.2.1 Ammonia Monitoring During the 10-Day Tests

After organisms are added to the testing chambers, ammonia in the surrogate chambers must be monitored to evaluate the potential for ammonia interference. The not-to-exceed toxicity threshold ammonia concentration for amphipods is 20 ppm total ammonia in pore water.

The not-to-exceed toxicity threshold ammonia concentration for mysids is established as the permissible concentration of un-ionized ammonia in test chamber *overlying* water and is related to the pH of the water (see Table 6-2).

**Table 6-2. Not-to-exceed Overlying Water Un-ionized Ammonia Threshold and pH Values for Solid Phase Testing Using *A. bahia***

| Ave. 10-Day pH | Not to Exceed Threshold[1] (ppm) |
|---|---|
| ≥ 8.1 | 0.5 |
| 7.9 – 8.0 | 0.4 |
| 7.7 – 7.8 | 0.3 |
| ≤ 7.6 | 0.2 |

[1]Toxicity thresholds based on Miller *et al* (1990), EPA (1994b), and unpublished NY/NJ Harbor dredged material testing data (1994-2005)

Daily measurements of pore water total ammonia (amphipods) and overlying water un-ionized ammonia (mysids, calculated using the dissociation model of Whitfield (1974) as programmed by Hampson (1977) or other suitable method) concentrations must be measured and recorded daily. Because ammonia toxicity to mysids increases as pH decreases, it is imperative that the pH in the overlying water in surrogate chambers is also measured daily.

The laboratory should inform NYD if ammonia levels during the test approach or exceed toxic threshold levels for mysids and amphipods. If overlying water un-ionized ammonia for mysids or porewater total ammonia for amphipods are found to exceed threshold levels during the 10 day test, once a day renewals of the overlying water should begin immediately in all chambers for that test organism. These renewals must be performed in all of the remaining surrogate chambers as well. Daily renewals must be discontinued as soon as non-toxic levels of porewater ammonia for amphipods and/or un-ionized overlying water ammonia for mysids is measured in the surrogate chambers. If the toxicity threshold values for either test organism are exceeded during any day of the test and there appears to be significant toxicity, contact NYD immediately to confirm the need for a retest using static renewal methods due to ammonia toxicity.

### 6.5.3 Test Conclusion

#### 6.5.3.1 Amphipod

At the end of the 10-day exposures, the surviving amphipods of each test, reference, and control testing chamber should be determined by carefully pouring the water and sediment through a 0.5-mm sieve (or other appropriate method established by the testing laboratory) and counting the amphipods retained on the surface of the sieve.

Debris sieved from amphipod test chambers should be saved in cups with salt water for 48 hours in the dark and checked for any amphipods not accounted for during post-test sieving and mortality recorded during the 10 day exposure period. Any amphipods remaining in the sediment debris should float to the surface of overlying water in the debris cup during this time.

If survival in reference sediment exposures is <80 percent NYD should be contacted immediately as the affected test(s) may have to be repeated. Delays in contacting NYD may lead to problems meeting holding times if a retest is required.

#### 6.5.3.2 Mysid

Surviving mysids should be removed from the test chambers at the end of the 10-day test by carefully decanting most of the overlying water (and mysids) through a 200 µm sieve while minimizing sediment disturbance/resuspension. This process should be repeated several times to ensure that all mysids are removed, overlying water should then be added to the test chamber slowly or using a squirt bottle filled with seawater and removed by decanting through the sieve. Water (and mysids) should be transferred immediately to a 2-L crystallization bowl or other suitable glass container for counting over a light table. Other effective procedures established by the testing laboratory may also be used to enumerate mysids at the end of the 10 day test period.

If survival in reference sediment exposures is <90 percent NYD should be contacted immediately as the affected test(s) may have to be repeated. Delays in contacting NYD may lead to problems meeting sediment holding times if a retest is required.

### 6.6 Results/Reporting/Statistical Analysis

One way unpaired t-tests ($\alpha = 0.05$) should be used to compare survivals in replicates of mysids and/or amphipods exposed to the test material to survivals in reference material replicates.

All testing, sampling, and organism handling activities associated with dredged material testing must be fully documented to ensure that solid phase toxicity data are defensible and verifiable. All raw biological and water quality data must be included in the final report, as well as hard copies of all statistical analysis performed on the data. A checklist for data which outlines the minimum requirements for preparation of a final biological report is included as Appendix G.

### 6.7 Test Acceptability/Quality Assurance

#### 6.7.1 Control Survival

The main test acceptability criterion for whole sediment toxicity tests is control survival. Control survival must be 90 percent or higher in the solid phase toxicity tests or the test must be repeated.

#### 6.7.2 Reference Sediment Survival

Since reference survival is used directly to determine LPC compliance in solid phase toxicity tests, its role is critical in determining the significance of toxicity. Based on historic data demonstrating consistently high survival of test organisms exposed to Mud Dump Site Reference Area sediment

and the narrative criterion that appropriate reference sediment supports high test organism survival, reference organism survival data is evaluated as part of the QA requirements of a project and a minimum test acceptability criterion must be observed.

Reference sediment mortality should not exceed 20 percent in amphipod solid phase testing or 10 percent in the mysid solid phase testing. Affected tests must be repeated unless survival in the test sediment is ≥ 80 percent for amphipods or ≥ 90 percent for mysids and no other quality control issues are documented. In this case, NYD may elect to use control survival as the criterion to validate the test data.

### 6.7.3 Reference Toxicant Testing

A reference toxicant test must be performed on each batch of organisms received from an outside supplier or monthly for all in-house cultures. This testing is conducted with water-only exposures (i.e., no sediment). The testing should include 96-h exposures of at least 20 organisms to a dilution series of 5 geometric concentrations and a control. $LC_{50}$ values are to be calculated from this data and compared to a reference toxicant control chart compiled for the species/supplier at the testing laboratory. Laboratories are to compile $LC_{50}$ values for reference toxicant exposures and generate a control chart for each test species after data for a particular reference toxicant have been generated using five independent tests using five different lots of organisms. $LC_{50}$ values for each subsequent lot of organisms are then compared to this control chart to determine acceptable sensitivity. Two standard deviations above and below the mean are established as the bounds of acceptability for reference toxicant test $LC_{50}$ values. A copy of the control chart (most recent 20 tests) and associated survival counts and water quality data must be provided with the test data.

An unacceptable reference toxicant test may indicate a problem with the test organisms, procedures used by the testing laboratory, laboratory contamination or other unidentified QC problem. A failed reference toxicant test may result in data that is not usable for making dredged material management decisions on the associated test sample(s). Other factors will be considered before a dredged material test is deemed invalid (on the basis of a failed reference toxicant test), including survival trends in test, including cropping effects in associated sediment testing (i.e. mortality of greater than 20% in control, reference, and control samples), acceptable test water quality, evidence of appropriate organism acclimation and handling, and evaluation of laboratory test procedures.

### 6.7.4 Other QA/QC Elements

- Control data files for all test organisms used in testing should be maintained as part of a laboratory's quality control program.

- Internal bioassay QC checks consist of taxonomic verification, proper handling of test organisms, water quality, acceptable control and reference survival, reference-toxicant testing, proper test procedures, and monitoring for potential laboratory, control, and reference sediment contamination. For a more detailed discussion of biological QA/QC for sediment

testing see Moore et al. 1994 and EPA 1994.

- NYD and EPA require that original data records and QC information for dredged material testing be maintained and archived for at least 3 years from the submission of the laboratory report and made available to NYD upon request.

**Table 6-3. Whole-Sediment Mysid Bioassays**

| Summary of Test Conditions and Test Acceptability Criteria for Acute Toxicity Tests with Mysids, *Americamysis bahia* | |
|---|---|
| Test type: | Static non-renewal (initiate static renewal methods if un-ionized ammonia in overlying water exceeds levels in Table 6-2) |
| 2. Test duration: | 10 days |
| 3. Temperature: | 20 ± 2 °C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 1 L |
| 7. Sediment-water ratio: | Add ~625 mL water to 200 cc (~175 mL) homogenized whole-sediment |
| 8. Age of test organisms: | 1-5 days; 24-hour range in age |
| 9. No. of organisms per test chamber: | 20 |
| 10. Treatment levels: | Control, reference and test sediment |
| 11. No. of replicates per treatment: | 5 |
| 12. No. of organisms per treatment: | 100 |
| 13. Feeding regime: | add up to 0.2 mL *Artemia* nauplii concentrate at least once a day during bioassay to prevent cannibalism |
| 14. Test chamber aeration: | Gently aerate all chambers using 1 mL pipette (approximately 100 bubbles/minute) |
| 15. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater |
| 16. Salinity: | 30 ± 2 ‰ |
| 17. pH: | 7.8 ± 0.5 (target) |
| 18. DO: | DO concentrations must not fall below 40% saturation in any chamber |
| 19. Endpoint: | Mortality - Record total number of live organisms at the end of the test |
| 20. Test acceptability criterion: | ≥ 90% mean survival in control organisms.<br>If mean reference AND mean test organism survivals are <90%, test may need to be repeated (see Section 6.7.2) |
| 21. Test method references: | ASTM (2011); Ferretti *et al* (2000); Miller *et al* (1990); EPA (1994b, 2002) |

**Table 6-4. Whole-Sediment Amphipod Bioassays**

| Summary of Test Conditions and Test Acceptability Criteria for Acute Toxicity Tests with Amphipods, *Ampelisca abdita*, *Rhepoxynius abronius*, *Eohaustorius estuarius* and *Leptocheirus plumulosus* | |
|---|---|
| 1. Test type: | Static Non-Renewal (Static renewal if porewater ammonia concentrations exceed 20 ppm during test) |
| 2. Test duration: | 10 days |
| 3. Temperature: | *A. abdita* 20 ± 2 °C; *R. abronius* 15 ± 2 °C; *E. estuarius* 15 ± 2 °C;<br>*L. plumulosus* 25 ± 2 °C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | Constant light (reference toxicant test can be conducted using a 16 hour light, 8 hour dark cycle). |
| 6. Test chamber size: | 1 L or one quart Mason Jars |
| 7. Sediment-water ratio: | Add ~625 mL water to 200 cc (~175 mL) homogenized whole-sediment |
| 8. Age of test organisms: | Using nested sieves:<br>*A. abdita* - subadults, 3-5 mm, retained on 0.7 mm sieve after passing through a 1.0 mm sieve<br>*R. abronius* - 3-5 mm, retained on 1.0 mm sieve with large individuals (≥ 5mm) excluded<br>*E. estuarius* - 3-5 mm, retained on 1.0 mm sieve with large individuals (≥ 5mm) excluded<br>*L. plumulosus* - subadults, 2-4 mm, retained on 0.5 mm sieve after passing through a 0.7 mm sieve |
| 9. No. of organisms per test chamber: | 20 |
| 10. Treatment levels: | Control, reference and test sediment |
| 11. No. of replicates per treatment: | 5 |
| 12. No. of organisms per treatment: | 100 |
| 13. Feeding regime: | Must not be fed during 10-day bioassay. |
| 14. Test solution aeration: | Gently aerate all chambers using 1 mL pipette (approximately 100 bubbles/minute). |
| 15. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater. |
| 16. Salinity: | *A. abdita* 28 ± 2 ‰<br>*R. abronius* 28 ± 2 ‰<br>*E. estuarius* 20 ± 2 ‰<br>*L. plumulosus* 20 ± 2 ‰ |
| 17. pH: | 7.8 ± 0.5 (target) |

| Summary of Test Conditions and Test Acceptability Criteria for Acute Toxicity Tests with Amphipods, *Ampelisca abdita*, *Rhepoxynius abronius*, *Eohaustorius estuarius* and *Leptocheirus plumulosus* | |
|---|---|
| 18. DO: | DO concentrations in each chamber should be maintained at ≥ 90% saturation. |
| 19. Endpoint: | Mortality - Record total number of live organisms at the end of the test. |
| 20. Test acceptability criterion: | ≥ 90% mean survival in control organisms.<br>If mean reference AND mean test organism survivals are <80%, test may need to be repeated (see Section 6.7.2) |
| 21. Test method references: | ASTM (2011); EPA (1994); Ferretti *et al* (2000) |

## 7.0 WATER COLUMN TOXICITY TESTING

All methods and procedures to be used in assessing the suspended phase toxicity of dredged material proposed for placement at the HARS should be detailed in the QA Plan. Written protocols should be referenced to assure that data quality objectives are met for each of the following test conditions:

- Consistent sensitivity (reference toxicant effects) and health (control survival) of test organisms;
- Acceptable water quality (temperature, salinity, pH, dissolved oxygen) as specified in test protocols; and
- Appropriate number of replicates and frequency of observations as specified in the test protocols.

### 7.1 Introduction

Water column suspended particulate phase (SPP) tests measure the acute toxicity of the dissolved and suspended portions of the dredged material that remain in the water column after discharge of the dredged material. SPP of the proposed dredged material must be tested by exposing a crustacean, a vertebrate (fish), and a zooplankton bivalve larva to a dilution series containing dissolved and suspended components of the proposed dredged material.

The results of the SPP toxicity tests are used to calculate the $LC_{50}$ (or $EC_{50}$) of the dredged material in the water column that will be used by NYD and EPA Region 2 to determine compliance with the Limiting Permissible Concentration (LPC).

*Modifications to any of these procedures and test conditions outlined in this section must be approved by EPA, Region 2 before they are incorporated as standard operating procedures.*

### 7.2 Species Selection

SPP toxicity tests involve exposing fish (*Menidia menidia, M. beryllina*, or *M*. *peninsulae*), crustaceans (*Americamysis bahia*) and zooplankton (bivalve larvae: *Mytilus edulis, M. galloprovincialis, Mercenaria mercenaria, Crassostrea virginica,* or *Mulinia lateralis*) to a dilution series containing dissolved and suspended components of the proposed dredged material. The species list and age requirements are shown in Table 7-1.

Each lot of organisms obtained from a commercial supplier must be taxonomically verified. All in-house cultures must be positively identified annually. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

**Table 7-1. Test Species for Water Column (SPP) Toxicity Evaluations**

| Test Species | Testing Age |
|---|---|
| CRUSTACEAN | |
| *Americamysis bahia* | 1-5 days old, age difference within batch <24 hours |
| VERTEBRATE (FISH) –One | |
| *Menidia beryllina** <br> *M. menidia** <br> *M. peninsulae* | 9-14 days old; age difference within batch < 24 hours |
| ZOOPLANKTON (BIVALVE)–One | |
| *Mytilus edulis** <br> *Mytilus galloprovincialis** <br> *Mercenaria mercenaria* <br> *Crassostrea virginica* <br> *Mulinia lateralis* | Embryos within 4 hours of fertilization |

**Preferred species*

The testing laboratory should have established a reference toxicity test record for any species selected for use in dredged material testing (five reference toxicant tests with results within laboratory acceptability limits of ± 2 standard deviations and five control samples with acceptable survival and/or development).

### 7.3 Organism Handling/Acclimation

All organisms used for testing of SPP must be acclimated to the SPP control/dilution water. The recommended acclimation period should be at least 48 hours but not less than 24 hours. Bivalves are the exception. Bivalves are typically shipped in moist seagrass or newspaper and can be induced to spawn without a formal acclimation period.

For mysids and *Menidia* spp, water temperature should not change by more than 3 $^{o}$C and salinity should not change by more than 3 ‰ over any 24 hour period. Overlying water should be renewed every 24 to 48 hours during acclimation.

Mysids and *Menidia* spp must be fed daily (approximately 100 *Artemia* nauplii per mysid) during acclimation. Care must be taken not to overfeed as it can cause unacceptable water quality conditions.

If mortality exceeds 10 percent over the entire acclimation and holding period, or 5 percent over the final 24 hours of acclimation, it is recommended that the affected lot of organisms be discarded and replaced with a new batch of organisms.

### 7.4 Preparation of 100% SPP Test Sample

The suspended particulate phase is prepared by sub-sampling approximately 1 L of the homogenized dredged material test sample and combining it with unfiltered dredging site water in a sediment-to-water ratio of 1:4 on a volume basis at room temperature. After the correct ratio is achieved, the mixture is stirred continuously for 30 minutes. After the 30-minute mixing period, the mixture is allowed to settle for 1 hour. The liquid and the material remaining in suspension after the settling period represents the "100%" suspended particulate phase. The supernatant is then carefully siphoned off, without disturbing the settled material. It is best to start the SPP tests immediately after preparation, however a 24-hour holding period is allowable (completely mix the supernatant before use in preparation of the test concentrations for the vertebrate and invertebrate testing).

For bivalve larvae SPP testing of very fine-grained dredged materials, it may be necessary to centrifuge the supernatant until the suspension is clear enough to allow for adequate resolution during microscopic examination at the end of the test. In these cases, a subsample of the 100% SPP may be centrifuged for 10 minutes @ 1000 g prior to its use in preparing test dilution series for the bivalve larvae SPP test. SPP prepared using centrifugation may only be used for the bivalve larval test. Alternatively, the supernatant of the SPP that settled overnight may be gently siphoned and used for testing with the bivalve larvae.

In cases where the salinity of the dredging site water is detrimental to the health of the test organism (too low), the dredging site water should be adjusted with commercially available sea salts until the target salinity is obtained. Deionized water may be used to dilute the site water should the salinity be too high. The salt-amended site water should be adjusted using dilute HCl or NaOH to its original pH. Salinity adjustments should be performed prior to preparation of the 100% SPP.

### 7.5 Preparation of the Bivalve Embryo Stock

A suspension of fertilized eggs is used in the preparation of the bivalve larvae SPP test treatment dilution series. Follow the ASTM (2004; E-724-98) protocol to prepare the stock larvae suspension. Gametes of 2 or more individuals of each sex should be pooled during preparation of stock to improve genetic diversity.

### 7.6 Suspended Particulate Phase (SPP) Test Procedures

#### 7.6.1 SPP Test Setup

A series of dilutions of the 100% SPP must be prepared in appropriately sized chambers using clean seawater or aged artificial seawater (the control/dilution water). The series must include 100%, 50%, and 10% suspended particulate treatments and a 0% suspended particulate treatment (100% dilution-water treatment) which also serves as a control. If high toxicity is expected, an additional dilution of 1% SPP should be included in the test design to ensure that the median lethal concentration is bracketed.

A minimum of three additional chambers should be set up for the bivalve test to determine initial stocking densities of bivalve larvae and to monitor for larvae development during the test. The additional chambers should be placed at the beginning, middle, and end of the control and test chambers to better characterize stocking densities and development.

A minimum of five replicates per treatment (dilution) are required for all test organisms. A total of 20 organisms per replicate are required for all species, except bivalve larvae. Each bivalve test (and larval monitoring) chamber should be stocked at 20-30 embryos/mL.

### 7.6.2 Test Conditions

Test condition summary tables for each species are included as Tables 7-2 through 7-4.
Daily water quality records must be kept for salinity, temperature, DO, and pH.

Mysids must be fed daily (approximately 100 *Artemia nauplii* per mysid) during testing as a precaution against cannibalism. *Menidia* sp. may be fed after 48 hours of testing by adding 0.2 mL of concentrated <24 hr-old *Artemia* to each test chamber. Care must be taken not to overfeed as it can cause unacceptable water quality conditions.

The number of surviving fish and mysids for each replicate must be recorded at 0, 24, 48, 72, and 96 h. Any observed sublethal effects, such as physical or behavioral anomalies must also be recorded. Temperature, pH, dissolved oxygen, and salinity must be measured and recorded daily in each chamber for SPP mysid and fish assays. For the bivalve larval tests, the frequency of these measurements should be at the beginning and end of testing (surrogate chambers are recommended to be added for taking these water quality measurements to minimize chance of contamination from water quality instruments).

Bivalve larvae initial densities must be recorded at the start of the test and final observations must be made of at least 100 organisms per replicate (if available) after 48 or 72 hours. If mortality in an exposure chamber prevents 100 embryos from being evaluated, observations should be made on a volume of SPP that would be expected to yield 100 embryos had mortality not occurred (based on controls or the pretest chambers stocking information). The test is terminated when 90% of the larvae in the 0% (control) treatment reach the prodissoconch I (D shape) stage of development.

### 7.6.3 Test Conclusion

At the conclusion of the 96-hour exposure, *Menidia* and mysid test chambers should be emptied into a glass bowl for counting over a light table or other established enumeration procedure. In addition to counting the number of surviving organisms, any observed sublethal effects, such as behavioral or physical abnormalities should be recorded.

Final observations for bivalve larvae should include survival, normal “D”-shaped shell, misshapen

shell, empty shell (no meat), and trocophore stage. Misshapen shells and trocophores are included as alive for the survival endpoint, however, only larvae with normal "D" shell shape should be characterized as "normally developed."

### 7.7 Results Reporting/Statistical Analysis

Median lethal concentrations ($LC_{50}$) and/or median effective concentrations ($EC_{50}$) must be calculated for measured endpoints in all SPP tests. Test concentrations must be established where at least one test concentration supports survival of at least 50%. A description of $LC_{50}$/$EC_{50}$ calculations can be found in the EPA Acute Toxicity Manual (EPA 2002). Graphical interpolation, probit, and trimmed Spearman-Karber methods are the most common ways to calculate $LC_{50}$ and $EC_{50}$ concentrations based on the assumptions of the data and organism survival trends. If survival and/or normal development is >50 percent in the "100" percent SPP, the $LC_{50}$ or $EC_{50}$ should be reported as >100 percent

All testing, sampling, and organism handling activities associated with dredged material testing must be fully documented to ensure that SPP toxicity data are defensible and verifiable. All raw biological and water quality data must be included in the final report, as well as hard copies of all statistical analysis performed on the data. A checklist for data which outlines the minimum requirements for preparation of a final biological report is included as Appendix G.

### 7.8 Test Acceptability/Quality Assurance

#### 7.8.1 Control Survival

Control (i.e., 0 % SPP) survival in all tests must be $\geq$90 percent or the test data are invalid and the test must be repeated. Also, normal development in control bivalve larvae must be $\geq$60 percent for mussel and clam larvae and $\geq$70 percent for oyster larvae or the test must be repeated.

#### 7.8.2 Reference Toxicant Testing

A reference toxicant test must be performed on each batch of organisms received from an outside supplier or monthly for all in-house cultures. The testing should include 96-h exposures of at least 20 organisms per concentration to a dilution series of 5 geometric concentrations and a control for mysids and *Menidia*. Bivalve reference toxicant tests should contain 20-30 embryos per mL and should be conducted for 48-72 hours. All reference toxicant tests should contain a dilution series that includes a minimum of five reference toxicant concentrations (in duplicate) plus a control (0%). $LC_{50}$/$EC_{50}$s are to be calculated from this data and compared to a reference toxicant control chart compiled for the species/supplier. Laboratories are to compile $LC_{50}$/$EC_{50}$ values for reference toxicant exposures and generate a control chart for each test species after data for a particular reference toxicant have been generated on at least five lots of organisms. $LC_{50}$/$EC_{50}$ values for each subsequent lot of organisms are then compared to this control chart to determine acceptable sensitivity. Two standard deviations above and below the mean are established as the bounds of

acceptability for reference toxicant test $LC_{50}$/$EC_{50}$ results. A copy of the control chart must be provided with the test data.

An unacceptable reference toxicant test may indicate a problem with the test organisms, procedures used by the testing laboratory, laboratory contamination or other unidentified QC problem. A failed reference toxicant test may result in determination of an invalid test for the associated test sample(s). Other factors will be considered before a test is deemed invalid (on the basis of a failed reference toxicant test), including survival trend deviations, water quality, organism acclimation, and/or laboratory test procedures.

### 7.8.3 Other QA/QC

- Control data files for all test organisms used in testing should be maintained as part of a laboratory's quality control program.

- Internal bioassay QC checks consist of taxonomic verification, proper handling of test organisms, water quality, acceptable control and reference survival, reference-toxicant testing, proper test procedures, and monitoring for potential laboratory, control, and reference sediment contamination. For a more detailed discussion of biological QA/QC for sediment testing see Moore et al. 1994 and EPA 1994.

- NYD and EPA require that original data records and QC information for dredged material testing be maintained and archived for at least 3 years from the submission of the laboratory report and made available to NYD upon request.

**Table 7-2. Water Column (SPP) Mysid Bioassays**

| Summary of Test Conditions and Test Acceptability Criteria for Acute Water Column Toxicity Tests with Mysids, *Americamysis bahia* | |
|---|---|
| 1. Test type: | Static non-renewal |
| 2. Test duration: | 96 hours |
| 3. Temperature: | 20 ± 2 °C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 600 mL minimum |
| 7. Test solution volume: | 400 mL minimum |
| 8. Age of test organisms: | 1-5 days; 24-hour range in age |
| 9. No. of organisms per test chamber: | 20 |
| 10. No. of replicates per concentration: | 5 |
| 11. No. of organisms per concentration: | 100 |
| 12. Feeding regime: | *Artemia* nauplii are made available while holding prior to the test; add 0.2 mL *Artemia* nauplii concentrate daily to each test chamber. |
| 13. Test solution aeration: | None, unless DO concentration falls below 4.0 mg/L; rate should not exceed 100 bubbles/min; if aeration is necessary, all chambers must receive the same treatment. |
| 14. Suspended particulate phase (SPP): | Use unfiltered near (sub)surface dredging site water. Dredged sediment and water are combined in a sediment-to-water ratio of 1:4 (volume/volume). |
| 15. SPP holding time: | Use within 24 hours |
| 16. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean natural seawater. |
| 17. Salinity: | 30 ± 2 ‰ |
| 18. pH: | 7.8 ± 0.5 (target) |
| 19. DO: | DO concentrations in each chamber must not fall below 40% saturation. |
| 20. Test concentrations: | Minimum of four suspended particulate treatment levels (%): 0 (control, diluent), 10, 50, 100 (1% if high toxicity expected). |
| 21. Endpoint: | Mortality ($LC_{50}$) - Record survivorship per replicate at 0, 24, 48, 72 and 96 hours. |
| 22. Test acceptability criterion: | ≥ 90% survival in control (i.e., 0% SPP) organisms |
| 23. Test method references: | ASTM (2002, 2003b); EPA (2002) |

**Table 7-3. Water Column (SPP) Fish Bioassays**

| Summary of Test Conditions and Test Acceptability Criteria for Acute Toxicity Tests with Silversides, *Menidia beryllina*, *M. menidia*, and *M. peninsulae*[a] | |
|---|---|
| 1. Test type: | Static non-renewal |
| 2. Test duration: | 96 hours |
| 3. Temperature: | 20 ± 2 °C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 250 mL minimum |
| 7. Test solution volume: | 200 mL minimum |
| 8. Age of test organisms: | 9-14 days; 24-hour range in age |
| 9. No. of organisms per test chamber: | 20 |
| 10. No. of replicates per concentration: | 5 |
| 11. No. of organisms per concentration: | 100 minimum |
| 12. Feeding regime: | *Artemia* nauplii are made available while holding prior to the test; 0.2 mL <24 hour old concentrated *Artemia* sp. to each testing chamber after 48 hours of testing. |
| 13. Test solution aeration: | None, unless DO concentration falls below 4.0 mg/L; rate should not exceed 100 bubbles/min; if aeration is necessary, all chambers must receive the same treatment. |
| 14. Suspended particulate phase (SPP): | Use unfiltered near (sub)surface phase (SPP): dredging site water. Dredged sediment and water are combined in a sediment-to-water ratio of 1:4 (volume/volume). |
| 15. SPP holding time: | Use within 24 hours of preparation |
| 16. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean natural seawater. |
| 17. Salinity: | 30% ± 2% |
| 18. pH: | 7.8 ± 0.5 (target) |
| 19. DO: | DO concentrations in each chamber must not fall below 40% saturation. |
| 20. Test concentrations: | Minimum of four suspended particulate treatment levels (%): 0 (control, diluent), 10, 50, 100 (1% if high toxicity expected). |
| 21. Endpoint: | Mortality ($LC_{50}$) - Record survivorship per replicate at 0, 24, 48, 72 and 96 hours. |
| 22. Test acceptability criterion: | ≥ 90% survival in control (i.e., 0% SPP) organisms |
| 23. Test method references: | ASTM (2002); ASTM (2003b); EPA (2002) |

[a] *Use M. peninsulae* only when other two species are unavailable

**Table 7-4. Water Column Bivalve Bioassays**

| Summary of Test Conditions and Test Acceptability Criteria for Acute Toxicity Tests with Bivalve Larvae, *Mytilus edulis*, *M.galloprovincialis, Mercenaria mercenaria*, *Crassostrea virginica* and *Mulinia lateralis* | |
|---|---|
| 1. Test type: | Static non-renewal |
| 2. Test duration: | 48-72 hours development into straight hinge prodissoconch larvae (D-shape stage) |
| 3. Temperature: | *M. edulis* 16 ± 2 °C; *M. galloprovincialis*, 16 ± 2 °C; *M. mercenaria* 25 ± 2 °C; *C. virginica* 25 ± 2 °C; *M. lateralis* 25 ± 2°C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 250 mL minimum |
| 7. Test solution volume: | 200 mL minimum |
| 8. Age of test organisms: | Use embryos within 4 hours of fertilization |
| 9. Density of organisms: | 20-30 embryos/mL |
| 10. No. of replicates per concentration: | 5 |
| 11. No. of organisms per test chamber: | $N = S ( V_s / V_t )$, where:<br>N = embryo density in test chamber<br>S = mean embryo density in stock suspension<br>$V_s$ = volume of stock added to test chamber<br>$V_t$ = total volume of test solution<br>Embryos per test chamber divided by embryos per mL equals mL of stock per chamber |
| 12. Feeding regime: | None during test as uneaten food might decrease DO and biological activity of some test materials, and the embryos/larvae can survive without feeding for 72+ hours |
| 13. Test solution aeration: | None, since bubbles can collect within larval mantle cavity. Aerate only if DO falls 60% saturation |
| 14. Suspended particulate phase (SPP): | Use unfiltered near (sub)surface dredging site water. Dredged sediment and water are combined in a sediment-to-water ratio of 1:4 (volume/volume). |
| 15. SPP Holding time: | Use within 24 hours of preparation |
| 16. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean natural seawater |
| 17. Salinity: | 30 ± 3 ‰ |
| 18. pH: | 7.8 ± 0.5 |
| 19. DO: | DO concentrations in each test chamber must be between 60 |

| | |
|---|---|
| | and 100% saturation at all times |
| 20. Test concentrations: | Minimum of four suspended particulate treatment levels (%): 0 (control, diluent), 10, 50, 100. |
| 21. Endpoint: | Mortality ($LC_{50}$) - mortality / abnormality ($EC_{50}$) |
| 22. Test acceptability criterion: | The number of embryos that result in live larvae with completely developed shells at the end of the control (i.e., 0 % SPP) test must be at least 70% of the initial number for oysters or 60% for mussels and clams, and at least 90% of all introduced embryos should be alive at the end of test. |
| 23. Test Method References: | ASTM (2004) |

## 8.0 BIOACCUMULATION TESTING

All methods and procedures to be used in assessing the solid phase bioaccumulation potential of dredged material proposed for placement at the HARS should be outlined step by step in the QA Plan. Written protocols should be referenced to assure that data quality objectives are met for each of the following test conditions:

- Consistent sensitivity (reference toxicant effects) and health (control survival) of test organisms;
- Acceptable water quality (temperature, salinity, pH, dissolved oxygen, ammonia, flow or renewal rate) as specified in test protocols; and
- Appropriate number of replicates and frequency of observations as specified in the test protocols.

### 8.1 Introduction

Bioaccumulation refers to the accumulation of contaminants in the tissues of an organism through any route, including respiration, ingestion, or direct contact with contaminated sediment or water. A burrowing polychaete worm and a deposit-feeding bivalve are the required test organisms for the dredged material bioaccumulation test. Test organisms are exposed for 28 days to the dredged material composite, reference sediment, and laboratory control sample. At the conclusion of the exposure and a brief holding period in water (to allow purging of ingested sediment), the organisms from all treatments are sacrificed and their tissues are sent to an analytical laboratory to measure contaminant residues accumulated during exposures.

The guidance manual: *Bedded Sediment Bioaccumulation Tests*, by Lee *et al* (1993), discusses bioaccumulation methodology in detail and may be followed on any matter that does not conflict with the Green Book and this guidance manual.

Modifications to any of the procedures and test conditions outlined in this section must be approved by EPA Region 2 before they are incorporated as standard operating procedures.

### 8.2 Species Selection

*Neanthes virens* and *Macoma nasuta* are the preferred test species for all NYD projects.
Table 8-1 lists the acceptable test species for bioaccumulation testing that are readily available from commercial suppliers. If preferred species are not available, then a request for a substitute species from the list below must be secured in writing from NYD. Details of suppliers contacted, date contacted and the reason for unavailability must be provided in the written request.

**Table 8-1. Bioaccumulation Test Species**

| POLYCHAETES (One) | |
|---|---|
| Scientific Name | Common Name |
| *Neanthes virens** | Sand worm |
| *Nephtys* spp. | Shimmy worm |
| BIVALVES (One) | |
| Scientific Name | Common Name |
| *Macoma nasuta** | Bent-nosed clam |
| *Tapes japonica* | Japanese clam |

*Must use this species whenever possible

Each lot of organisms obtained from a commercial supplier must be taxonomically verified. A citation of the taxonomic key used and the distinguishing characteristics must be documented during verification.

The testing laboratory should have established DOC for any species selected (including alternate species) for use in dredged material testing (five reference toxicant tests with results within laboratory acceptability limits of ± 2 standard deviations and five control samples with acceptable survival and/or development).

### 8.3 Organism Handling/Acclimation

Organisms should be acclimated a minimum of 48 hours prior to use in testing. Temperature should not change by more than 3 $^{o}$C and salinity should not change by more than 3 ‰ over any 24 hour period. *M. nasuta* should be held in flow through conditions whenever possible. Sand worms do not require feeding but *M. nasuta* should be fed daily during acclimation. If the clams are held under flow through conditions, then flow should be halted until the food has been filtered by the test organisms. Organisms must not be fed during the 28-day bioaccumulation testing.

Temperature, pH, salinity, and dissolved oxygen must be measured daily during acclimation. Water quality parameters before and after holding water renewal (if applicable) should be recorded. Feeding records should also be maintained.

At the beginning of testing, a sufficient number of clams and worms should be depurated by holding them in clean seawater for 24 hours without feeding for use as pretest or baseline tissue for chemical analysis. Organisms must not be depurated longer than 24 hours. Fecal material from worms must be siphoned twice during the 24-hour depuration period. Following this depuration period, tissues from the pretest organism should be handled as per the guidance in Section 8.5.3.

If mortality exceeds 10 percent over the entire acclimation and holding period, or 5 percent over the

final 24 hours of acclimation, then it is recommended that the affected lot of organisms be discarded and the acclimation be started with a new batch of organisms.

### 8.4 Sample Selection, Handling, and Preparation for Bioaccumulation Tests

#### 8.4.1 Control Sediment

The purpose of the control sediment is to document the acceptability of the test conditions and to help verify the health of the organisms during the test. Therefore, it is imperative that the control sediment have no discernible negative influence on the test organism.

Each testing laboratory must provide the appropriate control sediment(s). The sediment can be collected from uncontaminated sites where field-collected organisms were obtained. If the organisms are laboratory reared or purchased from a supplier, then the control can be the sediment in which organisms were shipped or cultured. Field-collected control sediments should be press sieved through a 2.0 mm (or smaller) mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in bioaccumulation testing. Control sediment handling procedures must be documented in the final testing report.

Excessive mortality in the control-sediment (>10 percent) indicates a problem with the test conditions, control sample, or with the organisms. However, the bioaccumulation test is not a lethality test and may or may not require retesting (see Test Acceptability/Quality Assurance, Section 8.7).

#### 8.4.2 Reference Sediment

Reference sediment should be press sieved a 2.0 mm (or smaller) mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in bioaccumulation testing. Reference sediment handling procedures must be documented and included in the raw data of the final report.

Reference sediment must not be held longer than eight weeks before it is used in testing. Reference sediment should be used in biological testing as soon as possible to obviate the need for sediment resampling in the event bioassays need to be repeated.

#### 8.4.3 Overlying (Control) Water

Test chamber overlying water should be the same water in which the test organisms are held prior to testing. Testing laboratories are responsible for collecting suitable water from the field or creating it using artificial sea salt formulations. If artificial sea salts are used, they must not contain EDTA or sodium thiosulfate, as they could affect contaminant availability in the test sample matrix. All seawater must be aerated (if stored in large quantities) at the laboratory and/or kept at 4 $^{o}$C. Water should not be held longer than 14 days before use in biological testing. Records must be maintained to document methods used to collect and/or prepare control water and conditions under which

control water is kept in the laboratory. Natural or artificial seawater used in bioaccumulation assays must be analyzed on an annual basis for the same suite of chemical parameters that are used in the tissue chemistry analysis.

### 8.4.4 Test Sediment

The dredged material sample should be press sieved through a 2.0 mm (or smaller) mesh stainless steel sieve to remove indigenous organisms and sediment debris prior to use in bioaccumulation testing. Homogenize the sediment until a consistent sediment color and texture are obtained. The sample should be tightly capped following sieving and homogenization and stored at 4 $^{o}$C until ready for use.

Test sediments for biological evaluation must be tested within 8 weeks of collection. Sample holding time starts as soon as the first sediment core contributing to a composite is collected. Test sediments should be used in biological testing as soon as possible to obviate the need for sediment resampling in the event bioaccumulation tests need to be repeated.

## 8.5 Test Procedures

### 8.5.1 Test Setup

Aquaria of 37 L (approx. 10 gallon) or larger are recommended for use in bioaccumulation tests. Aquaria should be cleaned before use in accordance with EPA (2002). Sediment should cover the entire bottom of the test chambers to a minimum of 5 cm; however, more sediment (7-11 cm) is recommended to provide adequate nutritional resources to sustain test organisms for 28 days and adequate pressure on the shells for burrowing of *Macoma nasuta*.

Five replicates are required for each test and reference sediment treatment, and three replicates are required for the control sediment in each test species. Therefore, a total of 26 tanks are required for evaluating a dredged material project that has been approved for testing as a single testing reach (i.e., composite).

At least 20 individuals of each species are required in each test chamber, although more may be necessary to provide sufficient tissue volume at the end of the test exposure to effectively conduct the required chemical tissue analyses. In determining the appropriate quantity, the loading factor per tank should be considered (ASTM 1996). If organisms fail to burrow within the first hour of being added to the tanks, they should be replaced.

### 8.5.2 Test Conditions

A summary of required test conditions are provided in Tables 8-2 and 8-3. Daily records must be recorded for salinity, temperature, DO, pH, flow rate (daily initial and final flow rate) or water renewal date and time, obvious mortalities, and any sublethal effects for each aquarium. If a static renewal

design is used (see below), the above water quality parameters must also be measured immediately before and after water renewals. Failure of organisms to burrow into the sediment or any other physical or behavioral abnormalities must also be recorded on a daily basis.

*Organisms must not be fed during the 28 days of testing.*

#### 8.5.2.1 Flow Through Versus Static Renewal Exposures

Although an automated flow through test design is preferred (6 volume changes per day), the bioaccumulation exposures may also be performed as a static renewal. Static renewal exposures require that 80% of water volume be changed every two days, however a Monday, Wednesday, and Friday scheme would also be acceptable as long as acceptable water quality is maintained. The test design and ultimate frequency of renewals must ensure that acceptable water quality is maintained (including managing ammonia levels) to minimize stress to test organisms (Ferraro 1990, Lee 1993). If automated flow through procedures are employed, then the flow to each tank must be measured daily and recorded, noting adjustments made to any tank (i.e. flow rate as found, flow rate as adjusted).

Acceptable Water Quality (Ammonia Management)

For flow through exposures, organisms can be added after an overnight settling period with the water flowing. Ammonia monitoring and aeration is not required for flow through exposures.

If static renewal procedures are used, sediment for porewater ammonia analysis must be subsampled from each of the 10 test tanks. Organisms must not be added to the test tanks unless porewater ammonia concentrations are below 60 mg/L. Eighty percent of the overlying water must be renewed each day until porewater ammonia concentrations are reduced to less than 60 mg/L. Once porewater concentrations are below 60 mg/L, water should be renewed a final time and the aquaria stocked with test organisms on the following day. Water is then to be renewed every two days until the 28-day exposure is completed. All tanks must be gently aerated for the duration of the bioaccumulation test and ammonia concentrations must be monitored daily in overlying water (not porewater) for the first week of the exposures to document water quality. If unacceptable water quality develops or ammonia concentrations are shown to be increasing, daily water renewals may be necessary for the duration of the test.

Renewal Procedures/Minimizing Stress to Organisms During Renewal

For static renewal design, overlying water removal and refilling must not cause stress to the organisms or disturb the sediment. Improper renewal techniques may result in invalidation of bioaccumulation tests regardless of final organism survival. Gravity siphoning or peristaltic pumps may be used to remove overlying water from tanks. Addition of water to the tanks must be accomplished as expeditiously as possible with no disturbance to the sediments or organisms. Water should be dispersed along the sides of the aquaria using low-flow funnels, tubing with pinch clamps,

water delivery manifolds modified for low flow, or small diameter siphons to control water flow during renewals. Care must be taken to ensure that renewal water is at the required temperature, dissolved oxygen concentration, pH, and salinity for the species being tested. Water quality parameters (see Tables 8-2 and 8-3) must be measured and recorded immediately before and after water renewals. Also, the times of water removal and water addition for each tank must be recorded. If the test is conducted as static renewal and there is unacceptable survival in the control, this may result in the need to repeat the test because stress from the renewals will be presumed, regardless of the other test acceptability criteria in Tables 8-2 and 8-3.

### 8.5.3 Test Conclusion

At the conclusion of the 28-day exposure, all test and reference organisms must be depurated for 24 hours by holding them in "clean seawater" (no sediment). Organisms (or soft tissues from each replicate and treatment) must be transferred into appropriate, labeled containers for shipment to the analytical laboratory (see Table 8-4). Organisms used in the control treatments are not analyzed for chemical constituents. However, the control organisms should be archived at the end of the test until after the tissue data has passed EPA’s QA review.

**Table 8-2. Worm Bioaccumulation Assays**

| Summary of Test Conditions and Test Acceptability Criteria for 28-day Bioaccumulation Tests with Sand Worms, *Neanthes virens* | |
|---|---|
| 1. Test type: | Flow-through, six volume exchanges per day<br>or static renewal, 80% water volume change every two days |
| 2. Test duration: | 28 days |
| 3. Temperature: | 20 ± 2 °C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 37 L (minimum of 20 L of water per tank) |
| 7. Sediment depth/ overlying water: | Minimum of 5 cm of sediment/tank; 7-11 cm recommended. 16 L overlying water minimum volume |
| 8. No. of organisms per test chamber: | 20 |
| 9. Treatment levels: | Control, reference and test sediment |
| 10. No. of replicates per treatment: | 3 control, 5 reference, 5 test |
| 11. No. of organisms per treatment: | 60 control, 100 reference, 100 test (contingent on tissue mass required to perform chemical analyses). |
| 12. Feeding regime: | None |
| 13. Test aeration: | Flow through: aerate only if DO falls below 4.0 mg/L. If aeration necessary, all tanks must be aerated.<br>Static renewal: aerate all aquaria |
| 14. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater. |
| 15. Salinity: | 30 ± 2 ‰ |
| 16. pH: | 7.8 ± 0.5 (target) |
| 17. DO: | DO concentrations in each chamber must not fall below 60% saturation |
| 18. Depuration | 24 hours in clean seawater at end of test |
| 19. Endpoint: | Measured tissue concentrations (no. of live organisms at end of test should be recorded) |
| 20. Test acceptability criterion: | If < 90% survival in control organisms, then determine whether:<br>a) there are adequate replicates to obtain sufficient statistical power;<br>b) there is adequate tissue for chemical analyses;<br>c) organisms stressed (e.g. reference toxicant results outside 2 standard deviations, failure to burrow, reduced sediment processing rate, evidence of disease);<br>d) there is contamination of the system;<br>e) the control sediment is contaminated; |

| | |
|---|---|
| | f ) there are other quality control problems.<br><br>If you answer “no” to 21a or 21b, or “yes” or “not sure” to 21c-f, bioaccumulation tests may have to be rerun. Consult with NYD immediately. |
| 21. Test Method References: | ASTM (2002b); Boese and Lee (1992); Lee *et al* (1993) |

**Table 8-3. Bivalve Bioaccumulation Assays**

| Summary of Test Conditions and Test Acceptability Criteria for 28-day Bioaccumulation Tests with Bent-nosed Clams, *Macoma nasuta* | |
|---|---|
| 1. Test type: | Flow-through, six volume exchanges per day<br>or static renewal, 80% water volume change every two days |
| 2. Test duration: | 28 days |
| 3. Temperature: | 12 -14 $^{o}$C |
| 4. Light quality: | Ambient laboratory illumination |
| 5. Photoperiod: | 16 hour light, 8 hour dark |
| 6. Test chamber size: | 37 L ((minimum of 20 L of water per tank) |
| 7. Sediment-Water Ratio: | Minimum of 5 cm of sediment/tank. 7-11 cm recommended, especially when sediment has high silt content, may need up to 11 cm of sediment/tank (minimum 16 L of overlying water) |
| 8. No. of organisms per test chamber: | 20 |
| 9. Treatment levels: | Control, reference and test sediment |
| 10. No. of replicates per treatment: | 3 control, 5 reference, 5 test |
| 11. No. of organisms per treatment: | 60 control, 100 reference, 100 test |
| 12. Feeding regime: | None |
| 13. Test aeration: | Flow through: Aerate if DO concentration falls below 4.0 mg/L<br>Static renewal: Aerate all aquaria |
| 14. Dilution water: | Modified GP2, Forty Fathoms®, or equivalent, artificial seawater prepared with Milli-Q® or equivalent DI water, or clean seawater. |
| 15. Salinity: | 30 ± 2 ‰ |
| 16. pH: | 7.8 ± 0.5 (target) |
| 17. DO: | DO concentrations in each chamber must not fall below 60% saturation. |
| 18. Depuration | 24 hours in clean seawater at end of test. |
| 19. Endpoint: | Measured tissue concentrations (no. of live organisms at end of test should be recorded) |

| | |
|---|---|
| 20. Test acceptability criterion: | If < 90% survival in control organisms, then determine whether<br>a) there are adequate replicates to obtain sufficient statistical power;<br>b) there is adequate tissue for chemical analyses;<br>c) organisms stressed (e.g. reference toxicant results outside 2 standard deviations, failure to burrow, reduced sediment processing rate, evidence of disease);<br>d) there is contamination of the system;<br>e) the control sediment is contaminated; or<br>f) there are other quality control problems, including improper renewal technique.<br><br>If you answer “no” to 21a or 21b, or “yes” or “not sure” to 21c-f, bioaccumulation tests may have to be rerun. Consult with NYD immediately. |
| 21. Test Method References: | ASTM (2002b); Boese and Lee (1992); Ferraro *et al* (1990) |

**Table 8-4. Storage and Preservation of Tissue Samples**

| Analyte | Sample Matrix | Volume Required/ Sample Container | Sample Preservation | Maximum Holding Time |
|---|---|---|---|---|
| Organic Compounds (Pesticides, PCBs, PAHs, Dioxins, Furans) | Tissue | 10 - 50g ≈ 4 oz. wide mouth amber glass with Teflon®-lined lid | ≤ -20°C; Keep in dark | 1 Year |
| Total Metals | Tissue | 1 - 50g ≈ 4 oz. wide mouth glass (amber optional) or plastic (HDPE) | ≤ -20°C Keep in dark optional | 1 year |

### 8.6 Data Reporting

All testing, sampling, and organism handling activities associated with dredged material testing must be fully documented to ensure that bioaccumulation test data are defensible and verifiable. All raw biological and water quality data must be included in the final report. A checklist for data which outlines the minimum requirements for preparation of a final biological report is included as Appendix G.

### 8.7 Test Acceptability/Quality Assurance

#### 8.7.1 Control Survival

NYD must be contacted immediately if control survival is <90 percent to determine whether the test data is acceptable or the test needs to be repeated. NYD and EPA may request supporting data from the laboratories, including reference toxicant test data, organism handling/acclimation data, water quality, and survival in other treatments (test and reference sediment). Laboratories may be required to perform chemical analyses of control sediment, water or tissue to ensure that there was no system contamination. NYD and EPA will use this information to establish that test organisms were healthy, stress-free, and/or not exposed to any other factors which may have compromised the objectives of a bioaccumulation assay. If any anomaly or QC issue is identified that is likely to have affected bioaccumulation uptake mechanics or organism health, a retest will be required. If elevated mortality occurs across treatments, an assessment will also be made to determine whether there is adequate test organism tissue remaining in relevant treatments to allow for chemical analysis of the tissue and statistical analysis of the results.

### 8.7.2 Pretest Tissue Contaminant Levels

Significantly elevated contaminant levels in tissues of organisms randomly selected (and held in clean seawater for 24 hours) prior to initiation of bioaccumulation testing (pre-test analyses) may justify retesting of the sediment. If an applicant suspects that pretest concentrations may have significantly contributed to high levels measured after 28 days, NYD should be consulted to determine if the test should be repeated. 28-day concentrations will not be adjusted in any way for any pretest contamination.

### 8.7.3 Reference Toxicant Testing

A reference toxicant test must be conducted on a subset of the organisms used in the bioaccumulation exposure. The testing should include 96-h exposures of at least 20 organisms per concentration to a dilution series of 5 geometric concentrations of reference toxicant concentrations plus a control (0%). This is a water-only exposure (no sediment) and artificial or clean natural seawater is used as the diluent. The concentrations should be selected so that the median effective concentration is bracketed. A reference toxicant test is conducted on each batch of organisms received from an outside supplier or monthly for laboratory reared organisms. Survival in the control sample must be ≥ 90 percent or the test should be repeated.

### 8.7.4 Other QA/QC

Test and reference sample survival, data trends (e.g., similar elevated mortality occurring across test, reference, and control exposures), reference toxicant data, water quality data, organism handling, improper renewal procedures and general test procedures are the major data elements used in determination of data usability.

## 9.0 PHYSICAL AND CHEMICAL TESTING

Physical and chemical analyses required to evaluate the various samples obtained in support of HARS dredged material evaluations are presented in this chapter. While methods are recommended for some of the analyses, this program is performance-based and alternative appropriate methods that meet the QC objectives may be used to complete the required analyses. NYD reserves the right to require additional analyses for specific projects.

It is strongly suggested that the applicant utilize laboratories which hold some form of accreditation, such as NELAP, TNI or ISO. These independent third party accrediting bodies assess laboratories to specific standards ensuring that there are management systems and quality systems in place while also assessing the technical capability of the analysts.

### 9.1 Physical Analysis of Sediment

Proposed dredged material core and composite samples, reference sediment, and control sediment(s) must be analyzed for grain size, total solids/percent moisture, specific gravity, bulk density, and Atterberg limits as per Table 9-1.

**Table 9-1. Physical Analyses Required for Sediment Samples Used for HARS Evaluation**

| Physical Parameter | Core (or stratum) | Composite Dredged Material | Reference Sediment | Control Sediment(s) |
|---|---|---|---|---|
| Grain size | ● | ● | ● | ● |
| Total solids<br>% moisture | ● | ● | ● | ● |
| Specific gravity | | ● | | |
| Bulk density | | ● | | |
| Atterberg limits | | ● | | |

#### 9.1.1 Grain Size Analysis

Grain size must be determined for each core (or each stratum within a core), test composite, reference sediment, and all control sediments used in testing described in this manual. The grain-size analysis must be conducted according to the methods described by ASTM D422-63 (2007) and reported as percentages and volumes within these general size classes: Sand: ≥ 75 µm diameter; Silt: < 75 µm and ≥ 39 µm diameter; Clay: < 39 µm diameter.

### 9.1.2 Total Solids/Percent Moisture

Total solids/percent moisture must be determined for each core (or each stratum per core), test composite, reference sediment, and all control sediments used in testing described in this manual.

Measurement of total solids/percent moisture is a gravimetric determination of the organic and inorganic material remaining in a sample after it has been dried at a specified temperature. Total solids and percent moisture should be measured as described by Plumb (1981) or APHA (2005). The values of the total solids generally are used to convert concentrations of contaminants from a wet-weight to a dry-weight basis.

### 9.1.3 Specific Gravity

Specific gravity must be determined for each test composite used in testing described in this manual. Specific gravity is the ratio of the mass of a given volume of material to an equal volume of distilled water at the same temperature and should be measured as described by Plumb (1981). The specific gravity of a dredged material sample helps to predict the behavior (i.e., dispersal and settling characteristics) of dredged material after disposal. Because the specific gravity analysis requires a dry sample, it is usually performed in conjunction with the total solids determination.

### 9.1.4 Bulk Density

Bulk density must be determined for each test composite used in testing described in this manual. Total solids/percent moisture must be determined for each core (or each stratum per core), test composite, reference sediment, and all control sediments used in testing described in this manual. Bulk density is the total mass (solids plus water) per unit of total volume of a sample at a given moisture condition (ASTM D4531-86, 2008). Bulk density of the sample should be reported as both wet and dry.

### 9.1.5 Atterberg Limits

Atterberg limits must be determined for each test composite used in testing described in this manual. In current engineering usage, the Atterberg limits (ASTM D 4318-05, 2005) usually refer only to the liquid limit, plastic limit, and in some references, the shrinkage limit of a soil or sediment sample. The liquid limit is the water content, in percent, of a soil (or sediment) at the arbitrarily defined boundary between the semi-liquid and plastic states. The plastic limit is the water content, in percent, of a soil (or sediment) at the boundary between the plastic and semi-solid states. The plasticity index is typically calculated from Atterberg limits and is the size of the range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

### 9.1.6 Quality Control for Sediment Physical Analyses

To assure the quality of physical data, NYD requires that one sample per batch of 1 to 20 sediment samples be analyzed in triplicate for each of the parameters listed in the sections above, with at least one set of triplicate analysis determinations made using the project sediment composite. The QC performance acceptance criteria for the physical analyses are provided in Appendix D.

### 9.2 Chemical Analyses (Sediment, Water, and Tissue)

The specific requirements for chemical analyses will be communicated via the testing checklist supplied to the applicant by NYD during the application process (see Box 3-2). The typical analytes required for sediment, water, and tissue samples generated in support of the evaluation of dredged material proposed for placement at the HARS are shown in Table 9-2. NYD may elect to modify the list of required analytes for any individual project and will be communicated to the applicant via the testing checklist.

The analytical method(s) chosen must be capable of providing quantitative results down to the required reporting limit. Required RLs for each analyte and matrix are presented in Appendix B). The RL represents the lowest concentration at which an analyte can be reliably measured. It is defined as the concentration of the lowest acceptable calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

**Table 9-2. Chemical Analytes Required for Samples Used for HARS Evaluation**

| Chemical Parameter | Water/Elutriate | Sediment | Tissue |
|---|---|---|---|
| Total organic content (TOC) | | ● | |
| Inorganics[1] | ● | ● | ● |
| Polycyclic aromatic hydrocarbons (PAHs)[2] | | ● | ● |
| Chlorinated pesticides[3] | ● | ● | ● |
| Polychlorinated biphenyls (PCBs)[4] | ● | ● | ● |
| Dioxins/furans[5] | | ● | ● |
| Other industrial chemicals[6] | | ● | ● |

[1]Inorganics include: arsenic (As); cadmium (Cd); chromium (Cr); copper (Cu); lead (Pb); mercury (Hg); nickel (Ni); silver (Ag); zinc (Zn). Analysis of arsenic is not required for water and elutriate samples.
[2]PAHs include: acenapthene; acenaphthylene; anthracene; fluorene; naphthalene; phenanthrene; benz(*a*)anthracene; benz(*a*)pyrene; benzo(*g,h,i*)perylene; benzo(*b*)fluoranthene; benzo(*k*)fluoranthene; chrysene; dibenzo(*a,h*)anthracene; fluoranthene;indeno(*1,2,3-c,d*)pyrene; pyrene
[3]Chlorinated pesticides include aldrin; α-chlordane; *trans* nonachlor; heptachlor; heptachlor epoxide; dieldrin, DDD (*p,p'-; o,p'*); DDE (*p,p'-, o,p'*);, DDT (*p,p'-, o,p'*); endosulfan I; endosulfan II; endosulfan sulfate
[4]PCBs must be analyzed using congener-specific quantitation methods. Congeners must include 8, 18, 28, 44, 49, 52, 66, 87, 101, 105, 118, 128, 138, 153, 170, 180, 183, 184, 187, 195, 206, 209.
[5]Dioxins/furans include the seventeen 2,3,7,8-substituted dioxins and furans
[6]1,4-dichlorobenzene is the only chemical in this class

#### 9.2.1 Chemical Analysis of Sediment

With the exception of total organic carbon (TOC) content, chemical analysis of proposed dredged sediments is typically required to be conducted only on aliquots of the composite dredged material sample used for biological testing.

#### 9.2.1.1 TOC Content

TOC content of sediment is a measure of the total amount of volatile and non-volatile organic material in a sediment sample. TOC content must be determined on all dredging sediment samples (including composites, cores and strata sub-samples), reference, and control samples. A high-temperature combustion, rather than chemical oxidation, method must be used to determine TOC. The QC performance acceptance criterion for TOC analysis is provided in Appendix D.

#### 9.2.1.2 Organic and Inorganic Contaminants

Applicants must conduct bulk sediment chemical analyses on aliquots of the sediment composites used in biological testing (see Table 9-2 for common analytes required). In certain circumstances, NYD may elect to require analysis of each core (or each stratum) for specific analytes (e.g., dioxins/furans). In addition, NYD reserves the right to require chemical analysis of reference and control sediments used in bioassay testing.

Sediment concentrations of: inorganics (metals) must be reported as µg/g dry weight; organic contaminants (except dioxins/furans) must be reported as ng/g dry weight; dioxins/furans must be reported as pg/g wet and dry weight. All concentrations should be reported to three significant figures.

### 9.2.2 Chemical Analysis of Water Samples (Site Water and Elutriate)

Applicants must conduct triplicate chemical analyses (see Table 9-2 for analytes typically required) on aliquots of the site water collected at each testing unit area specified in the testing checklist supplied by NYD and of an elutriate prepared by mixing dredged material with site water taken from each testing unit[6]. The elutriate should be prepared using the method described in the next paragraph.

To provide sufficient volume for analysis of the suite of required analytes typically required in HARS evaluations, the elutriate is prepared by sub-sampling the homogenized dredged material test sample and combining it with unfiltered dredging site water in a sediment-to-water ratio of 1:4 on a volume basis at room temperature. After the correct ratio is achieved, the mixture is stirred vigorously for 30 minutes with a magnetic stirrer. At 10-minute intervals the mixture is also stirred manually to ensure complete mixing. After the 30-minute mixing period, the mixture is allowed to settle for 1 hour. The supernatant is then carefully siphoned off, without disturbing the settled material and 0.45

[6] In some instances, NYD may require the applicant to conduct triplicate analyses of overlying water used for biological tests or on disposal site water.

micron-filtered.

Water concentrations of inorganics (metals) must be reported as µg/L. Water concentrations of organic contaminants (pesticides and PCBs) must be reported as ng/L. All concentrations should be reported to three significant figures.

### 9.2.3 Chemical Analysis of Tissue

Tissues of organisms of each test, reference, and pre-test replicate must be analyzed (after a 24-hour depuration period in which organisms, including pretest organisms, are held in sea water only). Sufficient sample size is required to achieve the RLs listed in Appendix B. Inability to meet the applicable RLs or to conduct the appropriate number of replicate analyses because of insufficient sample size could result in rejection of data.

Tissues of control animals should be archived (if not analyzed concurrently with the other samples) as NYD reserves the right to require their analysis at any time. Control organisms and any excess tissue samples must be kept frozen until six months after announcement of test results in a NYD Public Notice.

Pre-test tissue analyses are used to confirm that the bioaccumulation test organism sources are "clean."[7] Reference tissue concentrations must be below guidance values for all analytes of concern. If these pretest or reference tissue requirements are not met, it will be the responsibility of the laboratory or the private applicant to repeat the 28-day bioassay/bioaccumulation study. If there are any questions regarding the quality or validity of the bioaccumulation test due to pretest, control or reference analyte levels, please contact NYD before proceeding.

Tissue concentrations must be reported as wet and dry weight. Concentrations of inorganics must be reported as µg/g; organics (except dioxins/furans) must be reported as ng/g; and dioxins/furans must be reported as pg/g. All concentrations should be reported to three significant figures.

### 9.2.4 Quality Control - Chemistry

This section provides guidance to ensure the quality of the data collected during the analysis of dredged material testing samples. Quality-control (QC) analyses must be incorporated into all field and laboratory activities. All points in the sampling and analytical procedures where QC checks are required must be defined, and the frequency, types of checks, and acceptance /rejection criteria must be stated in the QA Plan. NYD requires chemistry QC checks for each sample matrix, i.e., site water, elutriate, sediment, and tissue. The required QC samples listed in the following sections are in addition to the instrumental QC (e.g., initial and continuing check samples, surrogates, internal standards) specified in the individual methods.

[7] If practicable, pretest tissue can be analyzed prior to conducting 28-day testing to obviate repeating the test(s) due to pretest tissue analyte levels above normal background concentrations.

Field Sampling

The quality of the data obtained should be ensured through

- Collecting representative samples
- Using appropriate sampling techniques
- Protecting or preserving the samples until they are analyzed

Sediment samples

- Procedural/method blank — one per batch of 1-20 samples
- Matrix spike (in triplicate) -- - representative sediment sample (i.e., reference/control sediment) fortified to 3 to 5x the reporting limit (RL) identified in Appendix B of this manual; one set per batch of 1-20 samples
- Laboratory control standard (LCS) or laboratory fortified blank (LFB) --- extraction vessel fortified to 3 to 5x the RL; one per batch of 1-20 samples
- Standard reference material (SRM) — one per batch of 1-20 samples; must be a sediment-based matrix. If feasible, certified or consensus values for the SRM should be > 3x RL.
- Surrogate spike – per sample (organics only)

Water samples

- Procedural/method blank — one per batch of 1-20 samples
- Matrix spike (in triplicate) -- a representative site-water sample fortified to 3 to 5x the RL; one set per batch of 1-20 samples
- LCS/LFB --- laboratory reagent water sample fortified to 3 to 5x the RL ; one per batch of 1-20 samples;
- SRM — one per batch of 1-20 samples (must be a sea water-based SRM or one made by the laboratory using reagent grade water fortified with 2.0% NaCl). If feasible, where an SRM is available, the certified or consensus values for the SRM should be >3x RL
- Surrogate spike – per sample (organics only)

Tissue samples

- Procedural/method blank — one per batch of 1-20 samples
- Matrix spike (in triplicate)-- representative tissue sample (pretest tissue of the *M. nasuta* or *N. virens*) fortified to 3 to 5x the RL; one set per batch of 1-20 samples
- LCS/LFB --- extraction vessel fortified to 3 to 5x the RL; one per batch of 1-20 samples
- SRM — Not required
- Surrogate spike – per sample (organics only)

### 9.3 Data Documentation and Reporting

The analytical activities described in this manual must be documented as described in section 9.3.1 to ensure that the applicant's data are defensible and verifiable. At a minimum, all raw and reduced data for all tests must be submitted for review. As described in the 40 CFR 792, raw data are ". . . any laboratory worksheets, records, memoranda notes, or exact copies thereof, that are the result of

original observations and activities or a study and are necessary for the reconstruction and evaluation of the report of that study." NYD requires that original data records and QC information for dredged-material testing be maintained and archived for at least 3 years from the submission of the laboratory report. These materials must be available to NYD upon request.

Clear summaries should be provided for all matrices and analytes detailing all Quality Control (QC) samples (calibrations, blanks, control spikes, matrix spikes, replicate analysis, SRM analysis, etc.), project sample batches, extraction and analysis dates, QC sample results, and applicable calculated performance criteria as specified in this manual. A checklist which outlines the minimum content requirements for final reporting of chemical analytical data is included as Appendix H. NYD reserves the right to request additional data beyond that specified in the checklist before determining the acceptability of submitted data for rendering regulatory decisions.

Analytical data packages require a signature of the laboratory manager, quality assurance officer or designated analyst verifying that the analytical data provided is valid.

### 9.3.1 Laboratory Data Reports and QC Summaries

The laboratory data reports and QC summaries should provide at a minimum:

1. Sample custody and tracking: laboratories should ensure that sample custody has been maintained by checking that custody seals are placed over coolers and in some instances over sample containers to ensure that samples are not tampered with during shipment; provide sample receipt forms (including but not limited to, the date(s) and time(s) samples are received at the laboratory and the cooler temperature); chain of custody (COC) records and laboratory sample login reports; ensure that the sample identification numbers on the summaries are used consistently throughout the project and are traceable to the chain-of-custody and sample run logs;

2. Project narrative: a detailed project narrative discussing compliance and noncompliance with all analytical and QC requirements listed in the RTM. Any non-conformances /anomalies with the established QC acceptance criteria, as specified in the RTM must be noted in the project narrative for each matrix tested. In addition, each non-conformance result should be identified with a data qualifier in the report;

3. Holding times, preservation/storage conditions: ensure that all holding times, preservation and storage conditions are met (see Tables 5-3, 5-4, 5-5, 5-6 and 8-4) Failure to adhere to established holding times, preservation and storage conditions may result in rejection of the data;

4. Standard/certified reference material (SRM) samples: the SRM must contain "relevant" levels for the majority of the analytes to be tested. They can either be certified or consensus values, or both. By “relevant," we mean levels that can properly establish the laboratory's

ability to recover the analytes in question (i.e., >3x the RL). The report summary must include the manufacturer and catalog number of the SRM, certificate of analysis of the SRM including the certified or consensus values and associated control range, the results of the SRM samples, and the calculated percent recovery for each analyte;

5. Triplicate matrix spike sample: unspiked sample results, spike level used (concentration in appropriate units e.g., ng/g), spiked sample results and the calculated percent recovery for each analyte;

6. Sample weights: sample weights should be included on the QC sample summary sheets;

7. Internal standards: a summary of all internal standard recoveries (those added prior to extraction) for all samples analyzed;

8. Surrogate standards: a summary of all surrogate standard recoveries (those added throughout the cleanup steps) for all samples analyzed;

9. Procedural blank (method blank): sample results for each analyte and the associated RL;

10. Sample handling: a copy of all internal laboratory logbook forms, (e.g., sample extraction log book, extract cleanup log book, instrument analysis logbook) used to document the handling of the sample throughout the laboratory should be included with the data package;

11. Initial and continuing calibration checks: equipment must be calibrated prior to analysis of any samples, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria. The criteria for initial and continuing calibration are specified in the performance and acceptance criteria below.

It should be noted that the specified QA/QC requirements in this plan represent the minimum requirements for any given analytical method. Additional requirements which are method-specific should also be followed, as long as the minimum requirements presented in this document have been met.

The results for the various QA/QC samples must be reviewed by laboratory personnel immediately following the analysis of each sample batch. These results should then be used to determine if the RTM control limits and acceptance criteria have been exceeded and if any corrective actions must be taken, before processing a subsequent sample batch. Control limits are numerical data criteria that, when exceeded, require specific corrective action by the laboratory before subsequent analyses proceed. The control limits and acceptance criteria along with the recommended frequency of analysis for each QA/QC element or sample type are summarized in Appendix D of this manual.

## 10.0 REFERENCES

40 CFR 136. Code of Federal Regulations, Volume 40, Part 136, Appendix B, Method 608 and 625.

APHA. 2005. *Standard Methods for the Analysis of Water and Waste Water*. 21st Ed. Washington, DC: American Public Health Association, American Water Works Association, Water Pollution Control Federation. 1368 pp.

ASTM 2011. Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates. In Annual Book of ASTM Standards, 2011, Vol 11.06. E1367-03 (2008) Philadelphia, PA.

ASTM. 2005. *Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils.* Annual Book of ASTM Standards, Vol. 04.08. D 4318-05. Philadelphia, PA: ASTM International.

ASTM. 2004. *Standard Guide for Conducting Static Acute Toxicity Tests Starting with Embryos of Four Species of Saltwater Bivalve Molluscs.* In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05 E 724-98, Philadelphia, PA, pp. 105 - 125.

ASTM. 2003a. Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates, E1391-03. Philadelphia, PA. 93 pp.

ASTM. 2003b. Standard Guide for Conducting Acute Toxicity Tests on Aqueous Effluents with Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E1192-97. Philadelphia, PA, pp. 371 – 384.

ASTM. 2002a. Standard Guide for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, E729-96. Philadelphia, PA., pp 135 – 156.

ASTM. 2002b. E1022-94. Standard Guide for Conducting Bioconcentration Tests with Fishes and Saltwater Bivalve Mollusks. In 2005 Annual Book of ASTM Standards Section 11 Water and Environmental Technology, Volume 11.05, Philadelphia, PA, pp. 237 – 254.

ASTM. 1993a. Standard Test Method for Specific Gravity of Soils In 1992 Annual Book of ASTM Standards, Volume (D854-92)

ASTM. D422-63, 2007. Standard Test Method for Particle-Size Analysis of Soils

Boese, B.L. and H. Lee II. 1992. Synthesis of Methods to Predict Bioaccumulation of Sediment Pollutants. U.S. Environmental Protection Agency, ERL-N Contribution No. N232, Newport, OR.

Bower, CE and T Holm-Hansen. 1980. A Salicylate-hypochlorite method for determining ammonia in seawater. *Can. J. Aquat. Sci.* 37:794-798.

EPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 5th ed. EPA-821-R-02-012. Office of Research and Development, Washington, D.C. 20460.

EPA. 1994. Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods. EPA 600/R-94/025 (June). Office of Research and Development, EPA, Narragansett, RI 02882

EPA. 1994b. Southerland, Elizabeth to Mario Del Vicario, June 14, 1994. "Recommendations for Conducting Sediment Toxicity Test with *Americamysis bahia* when Ammonia may be Present at Toxicity Levels"

EPA and USACE-NYD. 1992. Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal. USACE-NYD, Water Compliance Section.

EPA and USACE. 1991. Evaluation of Dredged Material for Ocean Disposal (Testing Manual). Environmental Protection Agency/U.S. Army Corps of Engineers. U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS. EPA-503/8-91/001

EPA and USACE. 1998. Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual (The Inland Testing Manual), Washington, DC. EPA-823-B-98-004.

Ferraro, SH, H Lee II, R Ozretich, and D Specht. 1990. Predicting bioaccumulation potential: A test of a fugacity-based model. *Arch Environ Contam Toxicol* 19:386-394.

Ferretti, JA, DF Calesso, JM Lazorchak, TJ Dolce, J Arnold, W Goodfellow, ME Smith, JR Serbst. 2004. Interlaboratory comparison of a reduced volume marine sediment toxicity test method using the amphipod *Ampelisca abdita*. *Environ Toxicol Chem* 23(3):632–637.

Ferretti, JA, DF Calesso, T Hermon. 2000. Evaluation of methods to remove ammonia interference in marine sediment toxicity tests. *Environ Toxicol Chem* 19 (8):1935-1941.

Hampson, BL 1977. The analysis of ammonia in polluted sea water. *Water Research* 11:305-308.

Lee, H. III, BL Boese, J Peltier, M Sinsor, DT Sprecht and RC Randall. 1993. Guidance Manual: Bedded Bioaccumulation Tests. ERL-N Contribution No. N111, EPA 600/R-93/183. Newport, OR.

Miller, DC, Poucher, S., Cardin, JC, and Hansen, D. 1990. The acute and chronic toxicity of ammonia to marine fish and a mysid. *Arch Environ Contam Toxicol* 19:40-48.

Moore, DW, TM Dillon, JQ Word and JA Ward. 1994. Quality Assurance/Quality Control (QA/QC) Guidance for Laboratory Dredged Material Bioassays. Results of QA/QC Workshop Held May 26-27, 1993, in Seattle, Washington. USACE, WES, Misc. Paper D-94-3, May 1994.

Plumb, RH, 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.

SAIC. 1993. Data Report #2: Sediment/Tissue Baseline Surveys New York Mud Dump Site Dioxin Capping Monitoring Project, November 1992. U.S. Army Corps of Engineers, New York District, New York, NY.

Whitfield, M. 1974. The hydrolysis of ammonia ions in seawater – a theoretical study. *J Mar Biol Assoc UK* 54:565-580.

# **APPENDIX A:** Standards for Submission of Geotechnical Information Used for Determination of Pleistocene Glacial Till and/or Red Clay

1. This memorandum specifically addresses those dredging projects where removal of Pleistocene glacial till and/or red clay with subsequent placement at the Historic Area Remediation Site (HARS) is proposed. The following standards and requirements have been developed to help ensure that geotechnical data submitted to NY District provides complete and unambiguous documentation of these project sediments.

2. These types of projects typically involve the submission of two sets of core data containing Pleistocene material:

a) Pre-dredging cores (PDCs) are used to determine the presence and depth of Pleistocene sediments within the dredging project footprint, and for general sediment characterization of the dredging prism.

b) Material Separation Plan cores (MSPCs) are used to determine if all (< 6 inches) non-HARS materials have been removed from the dredging footprint prior to dredging of HARS-suitable Pleistocene red clay and/or glacial till.

Please Note: Applicants should include any existing geotechnical data which documents the presence of undisturbed Pleistocene sediments within the dredging footprint with their application submission.

3. The geotechnical data and associated information described below must be collected and interpreted by an expert familiar with geotechnical data collection, interpretation, and documentation. All submissions must include the following:

A. **Maps** - All maps submitted must be legible, have a bar scale, a north arrow, and latitude-longitude coordinates plotted on the map. At a minimum, the NW and SE corners of the map must have latitude-longitude coordinates. Lines depicting the shoreline and other features (e.g. depth contours) must be thick enough to allow production of legible photocopies. All features must have labels and/or a map key.

When requesting a proposed sampling plan for PDCs, at least two maps must be submitted: a) one map showing the location of the project area within the general region (e.g. New York Harbor area), and b) a map, or maps, showing details of the project area. This detailed project area map must contain a clearly marked proposed dredging footprint along with bathymetric contours at 5 foot intervals, developed from the most recent hydrographic survey. Three copies of each map must be submitted to NY District. All proposed PDC locations must be depicted on the detailed project area map(s). These locations must be approved in writing by NYD prior to collection of any

PDCs. An example project map with PDC locations is shown in Figure 1.

When requesting a sampling plan for MSPCs, a detailed project area map containing the clearly marked permitted dredging footprint, along with bathymetric contours at 1 foot intervals developed from the most recent hydrographic surveys and plotted at a scale of 1" = 40 feet, must be submitted. NYD will use this map to indicate sampling locations for the MSPCs, and return to the permittee. The following maps must be submitted with the MSPC data:

- Post-environmental bucket dredging (first round) bathymetry, using 1-foot contours

- Difference map between the pre- and post-dredging bathymetry surveys, using 0.5-foot contours

If another round of environmental bucket dredging is required, another set of cores may also be required. A request for coring locations must be submitted to NY District along with the following maps:

- Post-environmental bucket dredging (second round) bathymetry, using 1-foot contours

- Difference map between the post-dredging surveys conducted after the first and second round of environmental bucket dredging, using 0.5-foot contours

B. **Cores** – PDCs must penetrate at least one foot deeper than the proposed dredging depth, to show the total thickness of sediments, and/or rock, which will be dredged. If vibracores cannot penetrate at least one foot below the proposed project depth, then a split-spoon sampler or other /coring device must be used to provide the required cores. MSPCs are taken after dredging of non-HARS material with an environmental bucket to determine the thickness of non-HARS material overlying HARS-suitable material. Cores must penetrate at least six inches into HARS-suitable material, to allow measurement of the overlying non-HARS sediment thickness. If use of a gravity coring device does not capture the required six-inches of HARS-suitable material additional weights, or some other modification, may be required to provide enough force to penetrate deep enough into the HARS-suitable material. If modification of a gravity coring device does not provide the required sampling of at least six-inches of HARS-suitable material within the core barrel/tube, use of a vibracore may be necessary to collect the required geotechnical data.

If vibracores are taken, cores must be split along the length of each core, such that two nearly identical halves are created, prior to photographing and logging (Figure 2). Core tubes should be cut and split such that minimal disruption of the sediments

inside of each core is caused during the cutting. The blade should be adjusted to only penetrate deep enough to ensure that only the core wall, or liner, is cut. After the core wall or liner is cut, a wire should be pulled through the sediments in the core, beginning at the bottom and pulling toward the top, resulting in two nearly identical core halves. Using a wire helps minimize disruption of the sediments contained within each core. Any samples collected from the cores should be taken from near the center, to avoid the disturbed sediment near the core edges. Similarly, the center of split spoon samples must be used for sampling, logging and photographing.

Equipment other than gravity cores, vibracores or split spoon samplers proposed for collecting sediment must be approved by the Corps prior to use.

Core logs must be of professional quality (typed, no hand-written field notes or illustrations). Each PDC and MSPC log must include the following information:

- latitude-longitude coordinates of the coring station
- elevation of the surface of the core referenced to MLW or other suitable local datum
- coring device used
- name of the person who logged the core
- date and time core was taken
- date core was logged
- elevation and depth scales along the entire length of each core
- blow counts and hammer weights for split-spoon and other percussion borings
- a graphic legend of the sediments/rocks found in each core
- locations in core where samples were extracted for analysis
- Munsell color of moist sample
- Material classification (sediment or rock type)
- remarks, such as odor (e.g., hydrogen sulfide, petroleum/chemical, etc.), visual stratification or lenses, debris, biological activity (e.g., burrows, living or dead organisms, shells, etc.), presence of oil sheen, vegetation (e.g., leaves, stems, peat, roots, etc.), any supplemental notes associated with each sediment or rock

In addition, PDCs must contain:

- shear strength values at depth intervals of four inches for all sediments contained within pre-dredging cores (a torvane shear meter may be used)

- grain size data of the Pleistocene material, including depths where grain size samples were collected*

** For PDCs, one sample of undisturbed Pleistocene sediment must be collected from each core for conventional sieve analysis (grain size analysis). Half-phi sieve intervals must be used. Grain-size distribution plotted as histograms derived from the sieve analysis data, along with the mean and standard deviation of the size distributions, must be presented with the other geotechnical data.*

Three copies of all core logs must be included with the data package. Graphic legends must use standard geological symbols (e,g., dots for sand, gray or black shading for mud, brick pattern for limestone, etc.). It is recommended that one core half be wrapped in plastic, to avoid drying out, and kept as an archive sample. The other core half can then be used for photographing, logging and sampling.

C. **Photographs** – High-resolution color photographs of each core must be taken. Photographs must clearly document the internal, near center, and undisturbed sediments of each core or split-spoon sample. The minimum print size is 8-inches by 10-inches. Three professional quality photo prints of all photographs must be submitted. Photographs must be taken such that no shadows are present across the exposed core surface. Indoor photography using uniform lighting, such as strobe units, is recommended. Relying on direct sunlight for core photographs provides poor, and likely unacceptable, results, due to variable sun angles and cloud cover causing shadows and non-uniform illumination. Photographs must be taken directly over the cores; no oblique angle photographs are allowed. The image quality should be sufficient to easily identify basic sediment types within the core/

Each photograph must be labeled with a legend that includes, at a minimum, the core identification number and date taken. A legible, proper photo scale must be included with each photograph, to allow accurate photo analysis. Tape measures must not be used for photo scales. Commercial, or precision printed scales, must be used (no hand-drawn scales) with half-inch or centimeter tick marks clearly visible in each photograph.

No more than 2 feet of core may occupy the long dimension of a photograph. Information documented by the photographs must match the information in the corresponding core log

2. A schematic example of a dredging project map with proposed PDC coring locations is provided in Figure 1. Note the clear outline of the shoreline, the proposed dredging footprint, the proposed PDC coring locations, and other requirements of the project map. A photograph of a properly split core is illustrated in Figure 2. Figure 3 is a sample core log. An example core photograph is provided in Figure 4.

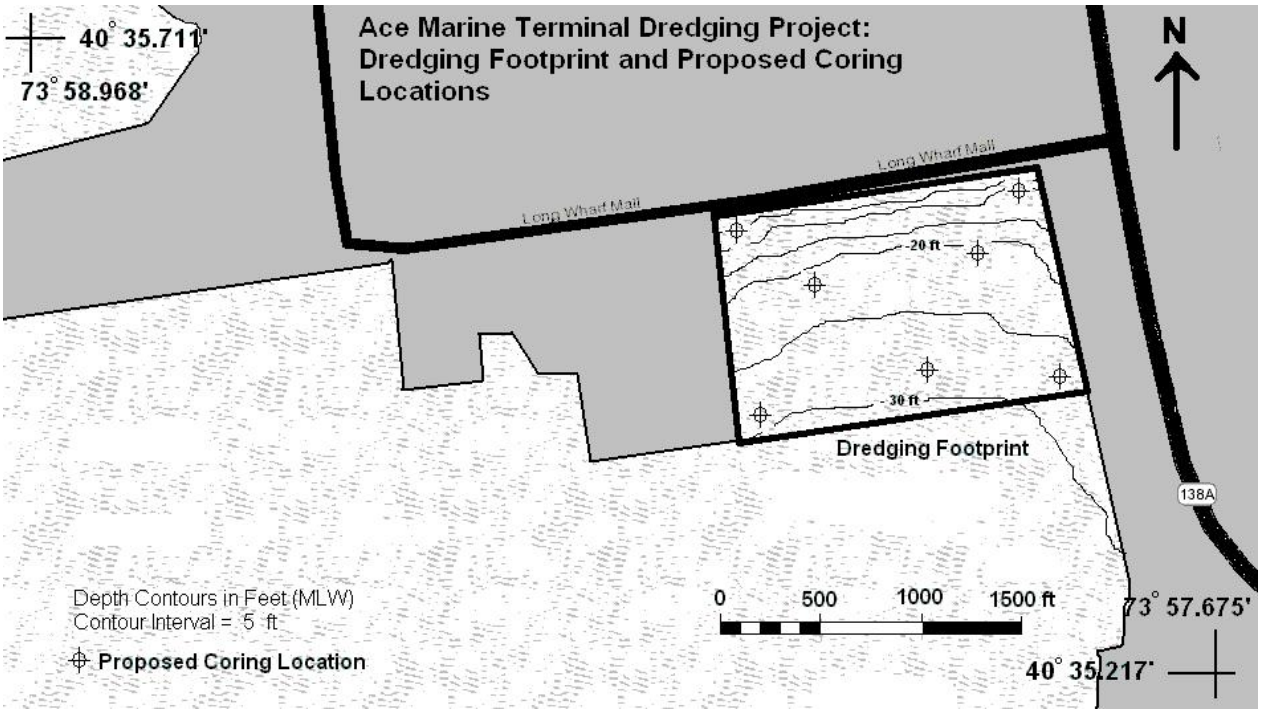



Figure 1. Example project map showing dredging project footprint, shoreline features, scale, north arrow, depth contours, latitude-longitude references, and proposed coring locations

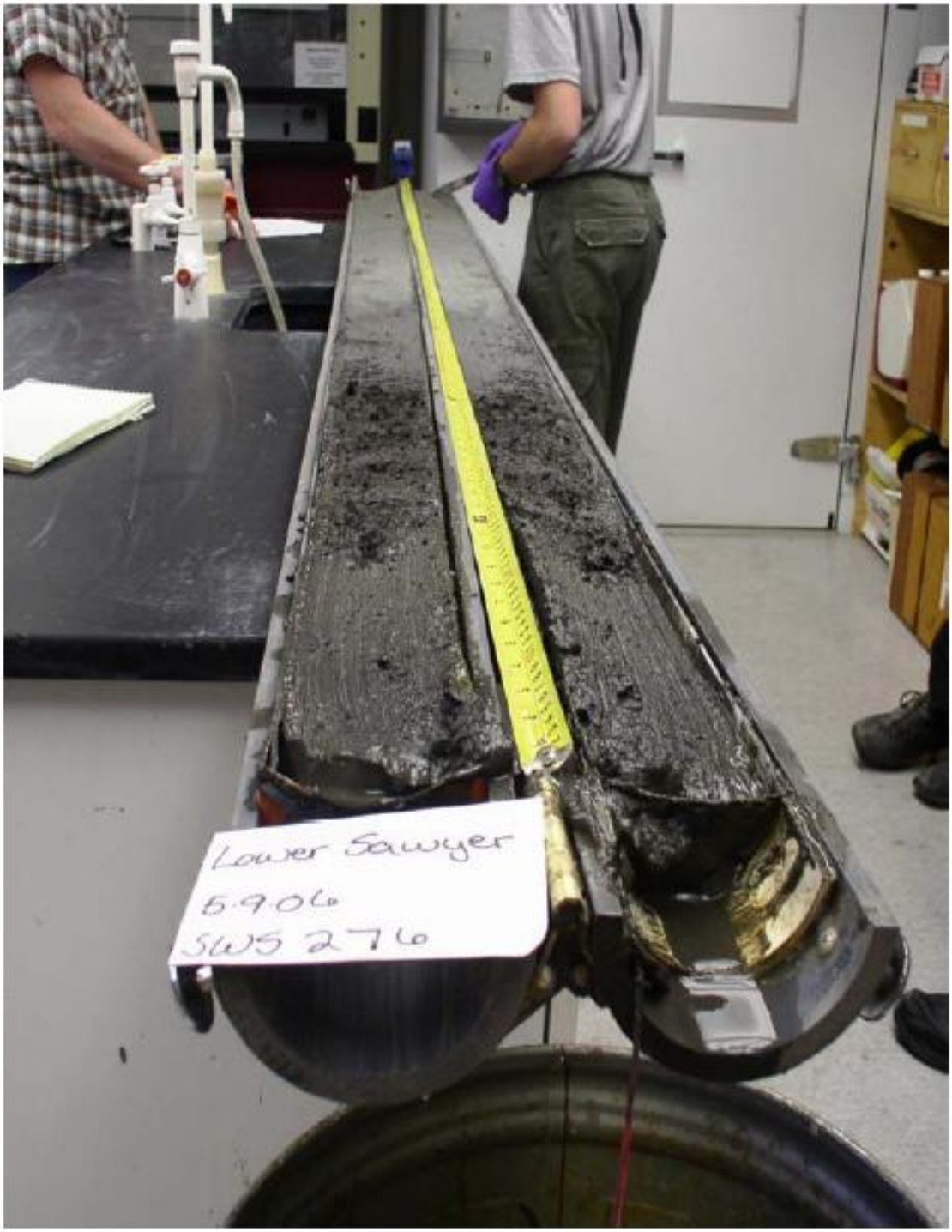



Figure 2. A properly split core, producing two nearly identical halves. The central, nearly undisturbed portions of either core half can be used for sample extraction. After splitting, photographs of one of the core halves can be taken, using an appropriate photo scale, and uniform lighting.

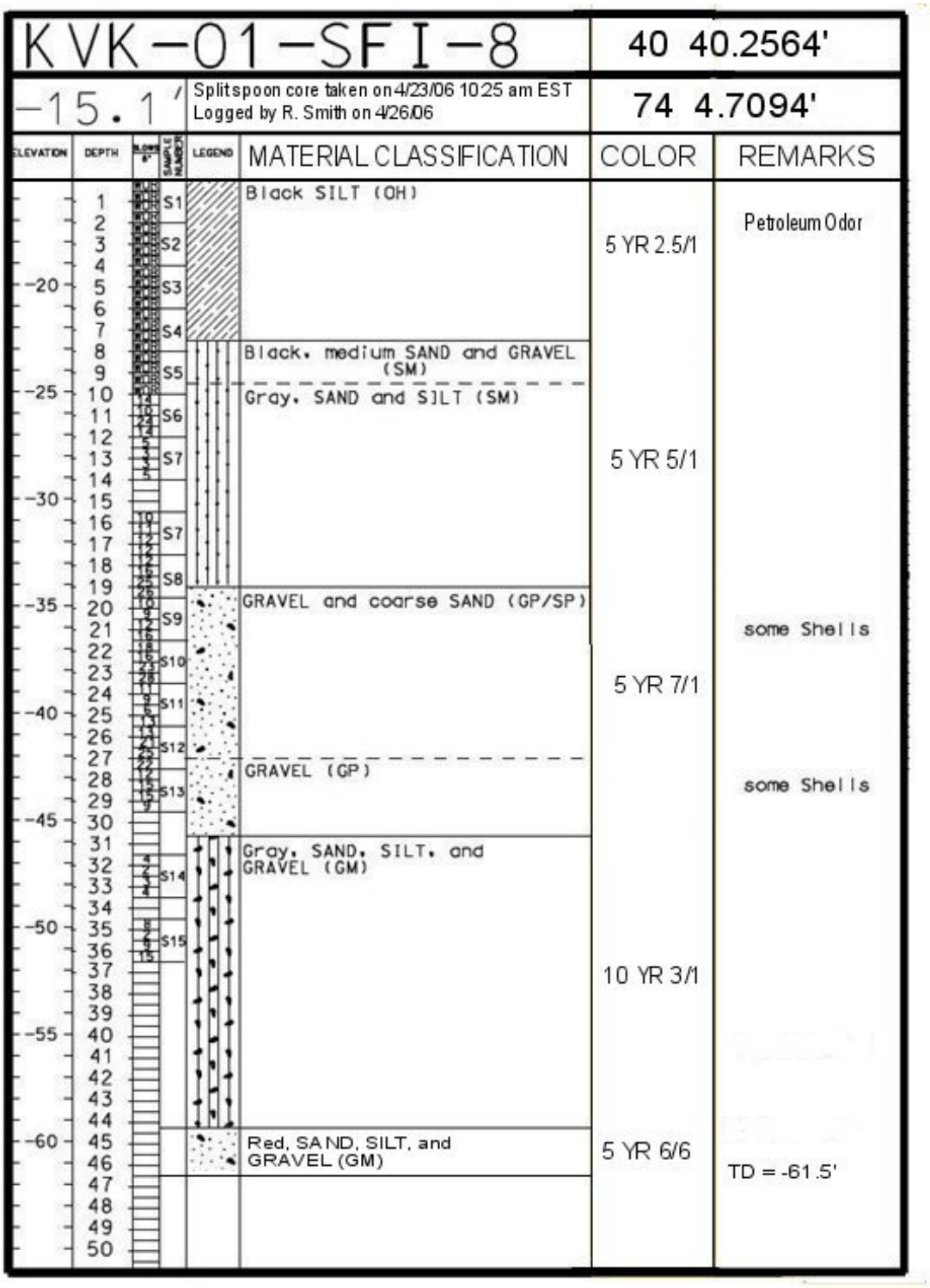



Figure 3. Example core log. All required information must be included on each core log.

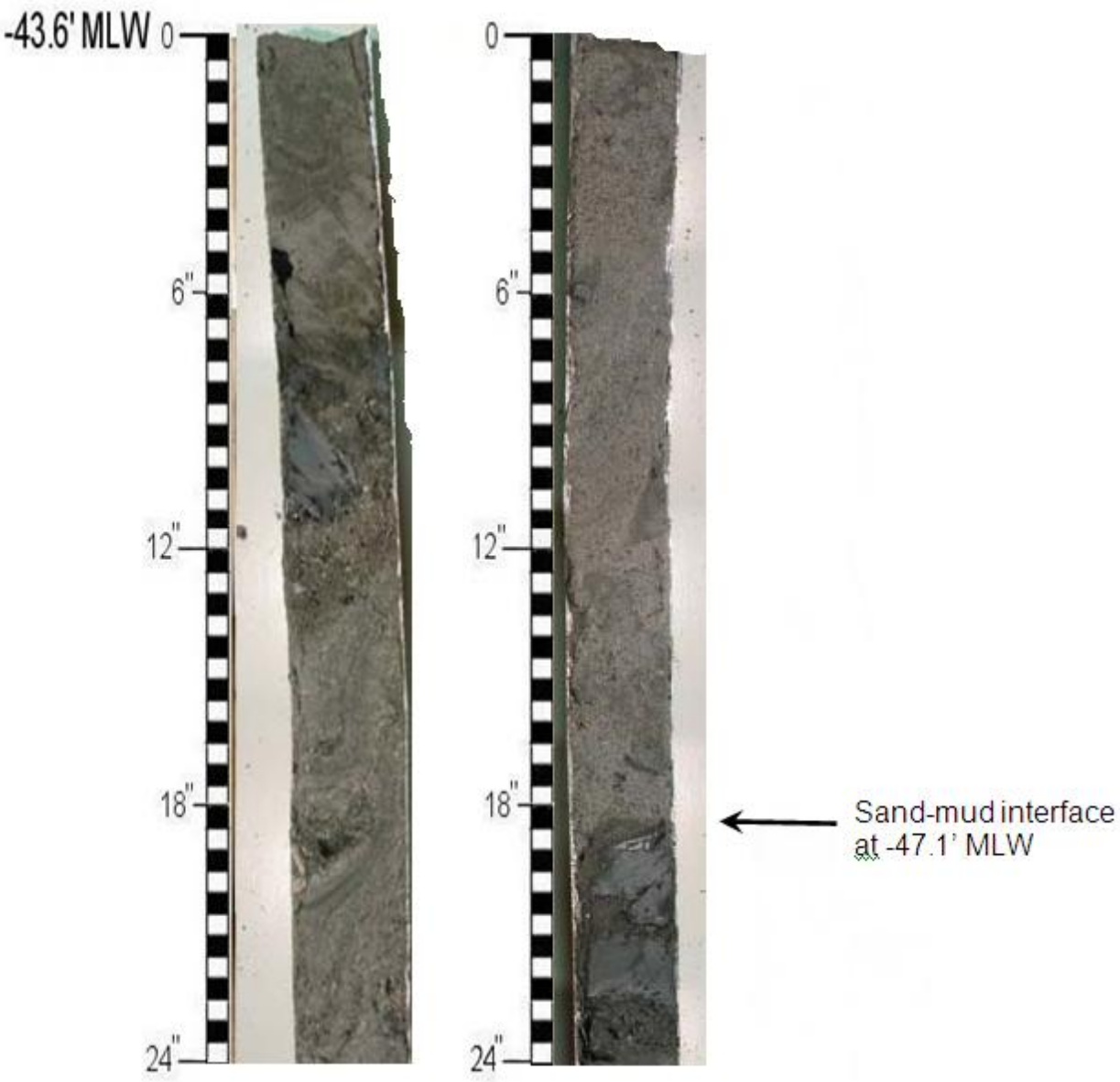



Figure 4. Example core photograph (vibra-core). Top of core must be located at the upper, left portion of photo, with deeper portions of the core down and to the right. No more than 24" of core must occupy the long dimension of each core photograph.

# **APPENDIX B:** Required Analytes and Reporting Limits

| TABLE 1<br>Constituent | Tissue<br>Reporting Limit (ng/g, wet) | Water<br>Reporting Limit (ng/L) | Sediment<br>Reporting Limit (ng/g, dry) |
|---|---|---|---|
| PAHs: | | | |
| Acenaphthene | 50 | N/R | 100 |
| Acenapthylene | 50 | N/R | 100 |
| Anthracene | 50 | N/R | 100 |
| Fluorene | 50 | N/R | 100 |
| Naphthalene | 50 | N/R | 100 |
| Phenanthrene | 50 | N/R | 100 |
| Benzo(a)anthracene* | 50 | N/R | 100 |
| Benzo(a)pyrene* | 50 | N/R | 100 |
| Benzo(g,h,i)perylene | 50 | N/R | 100 |
| Benzo(b)fluoranthene* | 50 | N/R | 100 |
| Benzo(k)fluoranthene* | 50 | N/R | 100 |
| Chrysene* | 50 | N/R | 100 |
| Dibenzo(a,h)anthracene* | 50 | N/R | 100 |
| Fluoranthene | 50 | N/R | 100 |
| Indeno(1,2,3-cd)pyrene* | 50 | N/R | 100 |
| Pyrene | 50 | N/R | 100 |
| 1,4-Dichlorobenzene | 3900 | N/R | 100 |

# **APPENDIX B (cont.):** Required Analytes and Reporting Limits

| TABLE 1<br>Constituent | Tissue Reporting Limit (ng/g, wet) | Water Reporting Limit (ug/L) | Sediment Reporting Limit (ng/g, dry) |
|---|---|---|---|
| Aldrin | 4.3 | 0.4 | 1 |
| Dieldrin | 1.5 | 0.20 | 1 |
| a-Chlordane | 2.7 | 0.030 | 1 |
| Trans nonachlor | 2.7 | 0.010 | 1 |
| Heptachlor | 2.7 | 0.010 | 1 |
| Heptachlor epoxide | 2.7 | 0.010 | 1 |
| Endosulfan I | 0.32 | 0.010 | 1 |
| Endosulfan II | 0.32 | 0.010 | 1 |
| Endosulfan sulfate | 0.32 | 0.010 | 1 |
| 4,4-DDT | 1 | 0.04 | 1 |
| 2,4-DDT | 1 | 0.010 | 1 |
| 4,4-DDD | 1 | 0.010 | 1 |
| 2,4-DDD | 1 | 0.010 | 1 |
| 4,4-DDE | 1 | 4.0 | 1 |
| 2,4-DDE | 1 | 0.010 | 1 |

# **APPENDIX B (cont.):** Required Analytes and Reporting Limits

| TABLE 1<br><br>Constituent | Tissue<br>Reporting Limit<br>(ng/g, wet) | Water<br>Reporting Limit<br>(ug/L) | Sediment<br>Reporting Limit<br>(ng/g, dry) |
|---|---|---|---|
| PCB 8 | 0.4 | 0.002 | 1 |
| PCB 18 | 0.4 | 0.002 | 1 |
| PCB 28 | 0.4 | 0.002 | 1 |
| PCB 44 | 0.4 | 0.002 | 1 |
| PCB 49 | 0.4 | 0.002 | 1 |
| PCB 52 | 0.4 | 0.002 | 1 |
| PCB 66 | 0.4 | 0.002 | 1 |
| PCB 87 | 0.4 | 0.002 | 1 |
| PCB 101 | 0.4 | 0.002 | 1 |
| PCB 105 | 0.4 | 0.002 | 1 |
| PCB 118 | 0.4 | 0.002 | 1 |
| PCB 128 | 0.4 | 0.002 | 1 |
| PCB 138 | 0.4 | 0.002 | 1 |
| PCB 153 | 0.4 | 0.002 | 1 |
| PCB 170 | 0.4 | 0.002 | 1 |
| PCB 180 | 0.4 | 0.002 | 1 |
| PCB 183 | 0.4 | 0.002 | 1 |
| PCB 184 | 0.4 | 0.002 | 1 |
| PCB 187 | 0.4 | 0.002 | 1 |
| PCB 195 | 0.4 | 0.002 | 1 |
| PCB 206 | 0.4 | 0.002 | 1 |
| PCB 209 | 0.4 | 0.002 | 1 |

## **APPENDIX B (cont.):** Required Analytes and Reporting Limits

| TABLE 1<br>Constituent | Tissue Reporting Limit (pg/g, wet) | Water Reporting Limit (ug/L) | Sediment Reporting Limit (pg/g, dry) |
|---|---|---|---|
| DIOXINS/FURANS: | | | |
| 2,3,7,8,-TCDD | 0.33 | N/R | 1 |
| 1,2,3,7,8-PeCDD | 1 | N/R | 2.5 |
| 1,2,3,4,7,8-HxCDD | 1 | N/R | 5 |
| 1,2,3,6,7,8-HxCDD | 1 | N/R | 5 |
| 1,2,3,7,8,9-HxCDD | 1 | N/R | 5 |
| 1,2,3,4,6,7,8-HpCDD | 10 | N/R | 5 |
| OCDD | 100 | N/R | 10 |
| 2,3,7,8-TCDF | 1 | N/R | 1 |
| 1,2,3,7,8-PeCDF | 1 | N/R | 2.5 |
| 2,3,4,7,8-PeCDF | 1 | N/R | 2.5 |
| 1,2,3,4,7,8-HxCDF | 1 | N/R | 5 |
| 1,2,3,6,7,8-HxCDF | 1 | N/R | 5 |
| 1,2,3,7,8,9-HxCDF | 1 | N/R | 5 |
| 2,3,4,6,7,8-HxCDF | 1 | N/R | 5 |
| 1,2,3,4,6,7,8-HpCDF | 10 | N/R | 5 |
| 1,2,3,4,7,8,9-HpCDF | 10 | N/R | 5 |
| OCDF | 100 | N/R | 10 |

## **APPENDIX B (cont.):** Required Analytes and Reporting Limits

| TABLE 1<br>Constituent | Tissue Reporting Limit (ug/g, wet) | Water Reporting Limit (ug/L) | Sediment Reporting Limit (ug/g, dry) |
|---|---|---|---|
| METALS: | | | |
| Arsenic | 4.2 | N/R | 1 |
| Cadmium | 0.10 | 14 | 1 |
| Chromium (total) | 3.9 | 350 | 1 |
| Copper | 3.2 | 1.0 | 1 |
| Lead | 0.43 | 70 | 1 |
| Mercury | 0.067 | 0.70 | 1 |
| Nickel | 1.3 | 25 | 1 |
| Silver | 0.47 | 0.80 | 1 |
| Zinc | 510 | 32 | 1 |

Footnotes:
N/R: not required

# **APPENDIX C:** HARS Placement Information Sheet

(This Appendix presents the content of the HARS Placement Information Sheet, an official copy of the form should be obtained from NYD)

**Date This Initial Sheet Completed by Potential Transport Permit Applicant:**

**INITIAL DATA SHEET FOR REQUESTING TRANSPORT PERMIT FOR DREDGED MATERIAL PLACEMENT IN ATLANTIC OCEAN (HARS)**

**[(HARS=USEPA-Designated Historic Area Remediation Site in the Atlantic Ocean Off NY/NJ Harbor. Transport of Dredged Materials to HARS site is subject to Section 103 of the Marine Protection, Research & Sanctuaries Act of 1972, as amended (33 U.S.C 1413)]**

**1. Permit Applicant/Potential Permit Applicant:**

**2. Application File Number (If already assigned by USACE):**

**3. Dredging Site Location: Latitude: Longitude:**

**4. Volumes of Sediments to be Dredged for Transport to Ocean Site (Including minimum required volume, overdepth volume, and total maximum volume):**

**5. Proposed Dredging Depths from Mean Lower Low Water (MLLW) Datum (Including minimum required dredging depth, overdepth dredging depth, and total maximum depth):**

**6. Size of Area(s) to be Dredged (Square Feet or Acres):**

**7. Locations of and Effluent Type For Any Outfall Pipes In or Adjacent to the Dredging Areas:**

**8. Planned Dredging Equipment (Clamshell Bucket Dredge, Hydraulic Pipeline Dredge, Excavator Dredge; etc.):**

**9. State Whether Intentional Scow/Barge Overflow Loading Is Planned:**

**10. Period of Time During Which Transport of Dredged Material to the Ocean Is Planned:**

**11. Dredging History of the Proposed Dredging Areas: (List any former USACE-issued permits, with permit number and date of permit issuance; as well as the last time dredging was performed in the area)**

# **APPENDIX D:** QA/QC – Chemical/Physical

Performance/Acceptance Criteria

**1. Metals** (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)

| QC Measurement | Frequency | Calculation | Control/Acceptance Criteria |
|---|---|---|---|
| Laboratory method blank | 1 per 20 samples | NA | No analyte should be detected at > RL |
| Matrix spikes (triplicate) | 1 set per 20 samples | % recovery and % RSD | 70 -130% (recovery) 30% (RSD) |
| Standard reference material (SRM) (sediment/water only) | 1 per 20 samples | % recovery *evaluated for analytes > 3x RL | 70 - 130% |
| LCS/LFB | 1 per 20 samples | % recovery | 70 -130% |
| Initial calibration check standards | Immediately following calibration curve | % recovery | 90 - 110% |
| Continuing calibration checks using standards | Minimum – check calibration at middle and end of each batch or 1 per 10 analyses, whichever is greater | % recovery | 90 - 110% from initial calibration for each analyte |

**2. Organics** (Pesticides, PCBs, PAHs)

| QC Measurement | Frequency | Calculation | Control/Acceptance Criteria |
|---|---|---|---|
| Laboratory method blank | 1 per 20 samples | NA | No analyte should be detected at > RL |
| Matrix spikes (triplicate) | 1 set per 20 samples | % recovery and % RSD | 50 - 150% (recovery) 50% (RSD) |
| Standard reference material (SRM) (sediment/water only) | 1 per 20 samples | % recovery<br><br>*evaluated for analytes >3x the RL | 50 - 150% |
| LCS/LFB | 1 per 20 samples | % recovery | 50 -150% |
| Surrogate Standards | Each sample | % recovery | 30-150%<br>* all applicable surrogate standards must be within the acceptance range to be considered acceptable |
| Initial calibration check standards | Immediately following calibration curve | % recovery | 80 – 120% |
| Continuing calibration checks using calibration standards | Minimum - middle and end of each batch or 1 per 10 analyses, whichever is greater. | % recovery | 80 – 120% from initial calibration for each analyte |

### 3. Dioxins/Furans

| QC Measurement | Frequency | Calculation | Control/Acceptance Criteria |
|---|---|---|---|
| Laboratory method blank | 1 per 20 samples | NA | No analyte should be detected at > RL |
| Matrix spikes (triplicate) | 1 set per 20 samples | % recovery and % RSD | 50 - 150% (recovery) 50% (RSD) |
| Standard reference material (SRM) (sediment only) | 1 per 20 samples | % recovery<br><br>*Evaluated for analytes >3x the RL | 50 - 150% |
| LCS/LFB | 1 per 20 samples | % recovery | 50 -150% |
| Internal standards (labeled compounds) | Each sample | % recovery | 25 - 150% |
| Initial calibration check standards | Immediately following calibration curve | % recovery | 80 – 120% for both unlabeled and labeled compounds. |
| Continuing Calibration Checks using calibration standards | Minimum - middle and end of each batch or 1 per 10 analyses, whichever is greater | % recovery | 80 – 120% from initial calibration for unlabeled compounds and 50 – 150% from initial calibration for labeled compounds |

### 4. TOC and Physical Parameters for Sediment

| Parameter | QC Measurement | Frequency | Calculation | Control/Acceptance Criteria |
|---|---|---|---|---|
| TOC<br>Grain size<br>Specific gravity<br>Bulk density | Triplicate | 1 triplicate per 20 samples (for each parameter) | % RSD | 20% |

# **APPENDIX E:** Demonstration of Capability Guidelines – Biological Testing

It is strongly recommended that applicants ensure that laboratories they use to perform sampling and testing of samples collected in support of dredged material projects in EPA Region 2 are capable of conducting the work described in this guidance manual. Ideally, the laboratories should perform a Demonstration of Capability (DOC) study prior to performing analysis of project samples to demonstrate that they are capable of conducting the work and to document the laboratories' current proficiency and capability. A successful DOC minimizes the potential for data unacceptability.

Region 2 has prepared the following guidelines to assist applicants (or their laboratories) in assessing their contractor's capability to conduct the testing outlined in this manual in accordance with the quality assurance and controls required by NYD and EPA Region 2. The applicant should request a signed acknowledgement (see section 4.0 of this Appendix for suggested format) that the laboratory has reviewed the DOC guidelines presented in this Appendix and that they have successfully conducted a DOC or work that is equivalent to that required in the DOC. Please note that this DOC guidance is provided for the convenience of the applicant and neither NYD nor EPA Region 2 will review/evaluate the DOC. Defined below are the procedures for a laboratory's self-acknowledgement of their DOC.

## 1.0 DOC Content

All laboratories performing analysis should have a quality system in place. Laboratories are responsible for the biological data they generate/report. Biological data packages used for this DOC require a signature of the laboratory manager, quality assurance officer and designated analyst verifying that the analytical data provided is valid. Please refer to the species lists in the RTM for the appropriate species to conduct the DOC. In the event the need to use a substitute species arises during an actual dredging project, data for potential alternate species must be submitted to NYD for review and approval.

The DOC for biological testing consists of two parts: reference toxicant data and control performance data. The acceptability and scope of data collected as part of the DOC are based on the performance criteria provided in the RTM (Regional Testing Manual). The elements of the two parts are summarized below (see Sections 2.0 and 3.0).

The biological data reports used for a laboratory DOC must include at a minimum:

- All $LC_{50}$/$EC_{50}$ calculations and/or percent control survival.

- A control chart for each test species with the $LC/EC_{50}$ endpoints plotted on the chart for all reference toxicant data.

- Raw bench sheets with water quality and daily test observations for every reference toxicant and control test provided as part of the DOC.

- A narrative statement that identifies and discusses any and all deviations from protocol

- Signature of the laboratory manager, quality assurance officer and designated analyst verifying that the analytical data provided is valid.

**2.0 Reference Toxicant Testing DOC Elements**

2.1 A minimum of five valid reference toxicant tests for each species used in the dredging program have been conducted. The tests have been conducted on a minimum of three different lots of each species tested.

2.2 Reference toxicant tests are water-only exposures for all species. Five test concentrations which bracket the $LC/EC_{50}$ value have been established for each test. All concentrations have been run in duplicate. The minimum control survival (90 percent) has been met to demonstrate test validity.

2.3 A control chart has been constructed which plots all of the data points for each species tested. The control chart shows the mean response and the ± 2 SD (standard deviation) lines on each control chart.

**3.0 Control Performance DOC Elements**

3.1 A minimum of five valid control exposures have been conducted for each species in the matrix used in the dredged material test. The control data from the reference toxicant portion of the DOC can be used for all water column dwelling organisms (silverside minnows, bivalve larvae).

3.2 Amphipods, mysids, and the bioaccumulation species (i.e. *N. virens* and *M. nasuta*) control data has been generated using sediment as outlined in the RTM.

3.2.1 It is recommended that the control sediment proposed for use in the dredged material program be used as part of the DOC.

3.2.2 Control survival must be equal to or greater than 90 percent.

## 4.0 DOC Self Disclosure Checklist

### A. Reference Toxicant Test Data

Yes or No : A minimum of five valid reference toxicant tests were conducted for each species proposed for use in the NYD and EPA Region 2 dredged material testing program

Comments:

Yes or No: A control chart for each unique species was assembled and is available for inspection upon request

Comments:

Yes or No: Control survival in all reference toxicant tests meet the test acceptability criterion as outlined in the RTM
Comments:

Any deviations from prescribed methods or other QC concerns and their potential impact on the validity of the data are discussed in a narrative statement

### B. Control Data

Yes or No : A minimum of 5 valid control data sets using the appropriate control matrix were conducted for each species proposed for use in the NYD and EPA Region 2 dredged material testing program
Comments:

Yes or No: Control survival in all tests submitted in all reference toxicant tests meet the test acceptability criterion as outlined in the RTM
Comments:

Any deviations from prescribed methods or other QC concerns and their potential impact on the validity of the data are discussed in a narrative statement

## 5.0 Demonstration of Capability Acknowledgement for Biological Testing

| | |
|---|---|
| Date: | |
| Laboratory name: | |
| Laboratory address: | |
| Laboratory scientist/technician name(s): | |

We the undersigned, acknowledge that:

1. All of the required analyses have been performed, documented, and meet the criteria of the DOC as set forth in Appendix E of the RTM.

2. The test method(s) was/were performed by the analyst(s) identified above.

3. Copies of the test method(s) and laboratory SOP(s) are available to all analysts on-site.

4. The data and documentation associated with this DOC are true, accurate, complete and self-explanatory[1].

5. All raw data and associated documentation including a copy of this form will be maintained at the laboratory performing the analysis in an organized manner and made available upon request for review by EPA or NYD.

| Laboratory Director's Name and Title | Signature | Date |
|---|---|---|
| Laboratory's Quality Assurance Officer | Signature | Date |

True: Consistent with supporting data and information.
Accurate: Based on good laboratory practices consistent with sound scientific principles and practices.
Complete: Includes the results and all supporting documentation
Self-Explanatory: The data and supporting documentation are properly labeled and the results are clear requiring no additional explanation.

# **APPENDIX F:** Demonstration of Capability (DOC) Guidelines – Chemical Testing

It is strongly recommended that applicants ensure that laboratories they use to perform testing of samples collected in support of dredged material projects in EPA Region 2 are capable of conducting the work described in this guidance manual. Ideally, the laboratories should perform a Demonstration of Capability (DOC) study prior to performing analysis of project samples to demonstrate that they are capable of conducting the work and to document the laboratories' current proficiency and capability. A successful DOC minimizes the potential for data unacceptability.

Region 2 has prepared the following guidelines to assist applicants (or their laboratories) in assessing their contractor's capability to conduct the testing outlined in this manual in accordance with the quality assurance and controls required by NYD and Region 2. The applicant should request a signed acknowledgement (see section 4.0 of this Appendix for suggested format) that the laboratory has reviewed the DOC guidelines presented in this Appendix and that they have successfully conducted a DOC or work that is equivalent to that required in the DOC. Please note that this DOC guidance is provided for the convenience of the applicant and neither NYD nor EPA Region 2 will review/evaluate the DOC. Defined below are the elements and procedures for a laboratory's self-acknowledgement of their DOC.

### 1.0 DOC Report Content

The DOC report should provide clear summaries for all matrices and analytes detailing all quality control (QC) samples (calibrations, blanks, control spikes, matrix spikes, replicate analysis, SRM analysis, etc.), project sample batches, extraction and analysis dates, QC sample results, and other applicable performance/acceptance criteria as specified in Appendix B of this manual.

The laboratory DOC data report and QC summary records should be maintained by the laboratory and, at a minimum, should include the following elements:

1.0 DOC narrative: a detailed narrative discussing compliance and noncompliance with all DOC elements and the associated performance criteria;

2.0 Laboratory method blank samples: Analysis of three blank samples processed through the entire procedure;

3.0 Laboratory control standard (LCS) or laboratory fortified blank (LFB) – Analysis of a spiked laboratory reagent water sample for water samples and a spiked extraction

vessel for solid samples (sediment/tissue) fortified to 3 to 5x the applicable RL listed in Appendix B of this manual. Spike level used (concentration) and the percent recovery for each analyte should be reported;

4.0 Matrix spike samples: Analysis of three replicate samples of a representative, relatively clean, sample (analyzed on consecutive or non-consecutive days) fortified to 3 to 5x the applicable RL listed in Appendix B of this manual. A representative sample for sediment should be a reference/control sediment/sand; a representative sample for tissue should be the unexposed, pre-test tissue of the *M. nasuta* or *N. virens*; a representative sample for water should be clean sea water or laboratory reagent water fortified with 2.0% NaCl. The study report is to include the unspiked sample results, the spiked sample results, the spike level used (concentration), the percent relative standard deviation and the percent recovery for each analyte;

5.0 Standard reference material (SRM) samples (sediment /water matrices only): Analysis of three representative SRM samples (analyzed on consecutive or non-consecutive days). The SRM should contain "relevant" levels for the majority of the analytes to be tested and has certified or consensus values. By “relevant", we mean levels that can properly establish the laboratory's ability to recover the analytes in question, i.e., ≥ 3x the RL identified in Appendix B of this manual. The study report should include the manufacturer and catalog number of the SRM, certificate of analysis including the certified or consensus values and associated control range, the results of the SRM samples, the standard deviation, the certified or consensus values and associated control range, and the percent difference from the certified or consensus values;

6.0 Internal standards (dioxins/furans only): a summary of the percent recoveries for all internal standards (labeled standards added prior to extraction; dioxins/furans only) for a representative set of at least 10 samples; and;

7.0 Surrogate standards (organics only): A summary of all surrogate standard recoveries (those added throughout the cleanup steps) for a representative set of at least 10 samples;

8.0 Initial and continuing calibration checks: a summary of the percent recovery for all calibration check samples analyzed for each analyte should be included in the DOC report.

All laboratories performing analysis should have and maintain a quality system in place. Laboratories are responsible for the analytical data they generate/report. Analytical data packages

used for this DOC require a signature of the laboratory manager or designated analyst verifying that the analytical data provided is valid.

**2.0 DOC Reporting Checklist** (to be completed for each matrix analyzed)

MATRIX (circle one): Sediment Tissue Water/Elutriate

Project Analytical Data Narrative – Discuss compliance and noncompliance with all analytical and QC requirements listed in the RTM. Provide a narrative for each of the following headings.

____Project name
____Parameter analyzed
____Name of laboratory (performing the analysis)
____Sample custody and processing
____QA/QC data quality indicators/objectives
____Analytical method summary (including any deviations, modifications or observations)
____Holding times are specified
____Method blanks
____Replicate precision
____Laboratory control sample
____Matrix spike (MS)
____Standard reference material (SRM)
____Data validation (statement and signature indicating that the data was reviewed/validated)
____Problems/corrective actions (if any)

Analytical Data Reporting
____Names of laboratories performing the analyses
____Name of laboratory contact
____Table of contents
____Raw and reduced data
____Sampling logs/field data sheets copies of log books etc.
____Sample tracking and traceability (from sample collection to disposal and/or archive)
____Initial and continuing calibration checks
____Data/QC summary tables

A) Table Headings
_____Laboratory name (performing the analyses)
_____Project name
_____Project number
_____Analyses and medium (e.g., metals in site water and elutriate)

_____For tissue data, specify type (*M. nasuta* or *N. virens*)
_____Sample Identification Number (traceable to the COC documentation. Samples must be traceable from laboratory to laboratory using the same sample ID number)
_____Date sample was prepared/extracted and analyzed
_____Summary of sample weights

B) Reports results using the appropriate units.
____Metals in tissue (ug/g) wet weight and dry weight
____Metals in water (ug/L)
____Metals in sediment (ug/g) dry weight
____Organics in tissue (ng/g) wet weight and dry weight
____Organics in water (ug/L)
____Organics in sediment (ng/g) dry weight
____Dioxins/furans in tissue (pg/g) wet weight and dry weight
____Dioxins/furans in sediment (ng/g) dry weight

C) Quality Assurance and Quality Control
1) Matrix spike triplicate samples analyzed
____Sample weight/volume
____Unspiked sample results
____Spiked sample results
____Concentration of spike level used and amount of spike added
____% recovery for each analyte

2) Standard reference material analyzed (if applicable)
____Sample weight/volume
____SRM contains relevant analytes
____Manufacturer and catalog # of SRM
____Copy of Certificate of Analysis (certified or consensus value)
____Results of the SRM Samples
____% recovery calculated for each analyte

3) Internal/Surrogate Standard Analyzed
____Results reported for each sample analyzed
____% recovery for each internal/surrogate standard

## 3.0 Demonstration of Capability Acknowledgment for Chemical Testing

| | |
|---|---|
| Date: | |
| Laboratory name: | |
| Laboratory address: | |
| Laboratory scientist/technician name(s): | |

Matrix:

Method Number, SOP# and Rev. #:

Analyte or class of analytes:

We the undersigned, acknowledge that:

1. All of the required analyses have been performed, documented, and meet the criteria of the DOC as set forth in Appendix F of the RTM.

2. The test method(s) was/were performed by the analyst(s) identified above on this certification.

3. Copies of the test method(s) and laboratory SOP(s) are available to all analysts on-site.

4. The data and documentation associated with this DOC are true, accurate, complete and self-explanatory[1].

5. All raw data and associated documentation including a copy of this form will be maintained at the laboratory performing the analysis in an organized manner and made available upon request for review by EPA or NYD.

| Laboratory Director's Name and Title | Signature | Date |
|---|---|---|
| Laboratory's Quality Assurance Officer | Signature | Date |

True: Consistent with supporting data and information.
Accurate: Based on good laboratory practices consistent with sound scientific principles and practices.
Complete: Includes the results and all supporting documentation
Self-Explanatory: The data and supporting documentation are properly labeled and the results are clear requiring no additional explanation.

# **APPENDIX G:** Checklist –Biological Data

FORMAT AND CONTENT FOR BIOLOGICAL DATA PACKAGES
NYD AND EPA REGION 2 DREDGED MATERIAL PROGRAM

### 1.0. Summary Tabular Data and Project Narrative

TEST PERFORMANCE SUMMARY
A summary table listing the percent survival in all control, reference, and test samples.

STATISTICAL ANALYSIS SUMMARY
A summary table containing the $LC/EC_{50}$ values for the suspended particulate phase (SPP) tests and t-tests from the solid phase tests

DEVIATIONS/QUALIFIER PAGE
A narrative which summarizes all of the deviations from the Green Book and Regional Guidance Manual protocols. Deviations of sample handling, test conditions, ammonia purging procedures, control performance, reference toxicant test performance, organism handling/acclimation, and water quality parameters should be provided in this section.

SAMPLING SUMMARY
A summary table which documents collection dates and holding times for the test, control, and reference sediment samples. Holding times for site water, SPP, and lab saltwater for all tests should be included in this table.

REPORT NARRATIVE
The data narrative should describe the major biological project activities and results. Computerized tables of results, water quality, and other pertinent information should be placed in this portion of the biological data package.

## 2.0 General Data Reporting Requirements

| | |
|---|---|
| RAW BIOLOGICAL AND WATER QUALITY DATA FROM TESTS | |
| | Survival and bivalve development data |
| | Water quality parameters |
| | Feeding schedule and amount (if applicable) |
| | Organism observations |
| | Summary of test conditions |
| | Project notebook/log entries and deviations from protocol |
| TEST ORGANISM HOLDING, HANDLING AND ACCLIMATION | |
| | Organism shipping data sheet provided by supplier |
| | Copy of overnight shipping airbill (if applicable) |
| | Internal receiving and distribution data |
| | Holding/acclimation records (incl. water quality, renewals, and feeding) |
| | Mortality during holding and acclimation |
| | Taxonomic identification for each species |
| | Feeding records |
| REFERENCE TOXICANT DATA | |
| | Raw bench sheets for reference toxicant tests |
| | Reference toxicant stock & test solution preparation sheet |
| | $LC/EC_{50}$ statistical calculations |
| | Updated reference toxicant control charts w/ acceptability limits |
| STATISTICAL DATA FROM DREDGED MATERIAL TESTS | |
| | Provide all computer generated $LC_{50}$, $EC_{50}$ and/or t-test spreadsheets or graphical interpolations for the SPP, solid phase tests, and t-tests comparing test and reference tissue chemistry. |
| INVALID TEST DATA | |
| | If a test was repeated for any reason, then the data from the original test must be included in the final report. If a serious deviation occurs which has the potential to affect test acceptability, then NYD and EPA Region 2 must be contacted immediately to determine if a retest is needed (to avoid missing established holding times) |

**3.0 Test Specific Information (additional to items specified in 2.0)**

| | |
|---|---|
| | AMPHIPOD SOLID PHASE TEST |
| | Pretest overlying water renewal log and total porewater ammonia data |
| | Total/un-ionized porewater ammonia measured in surrogate jars during testing |
| | |
| | MYSID SOLID PHASE TEST |
| | Pretest overlying water renewal log and total porewater ammonia data |
| | Total/un-ionized overlying un-ionized ammonia measured during testing |
| | |
| | SUSPENDED PARTICULATE PHASE TESTS (SPP) |
| | SPP preparation log (all volumes, mixing times, centrifuge info. etc.) |
| | Raw data for bivalve gamete collection and preparation |
| | |
| | BIOACCUMULATION TESTING |
| | Pretest ammonia purging data (Static Renewal) |
| | Flow calibration log – Initial and final adjusted flows (Flow-throughs) |
| | Dates and times of water removals and renewals (Static Renewal) |
| | |
| | Description of Siphon and Renewal Techniques (Static Renewal) |
| | Pre- and post-test depuration logs – time started/ended and flow rates |
| | Receiving logs for all natural saltwater (if collected) |
| | Preparation logs for all artificial saltwater |
| | If control survival <90%, provide DETAILED narrative for the 5 factors |
| | Raw statistical data comparing test and reference tissue chemistry |
| | |
| | SAMPLING / SAMPLE HANDLING |
| | Chain of custody forms for all test, control, and reference samples |
| | Field data sheets and/or sampling Logs (incl. photos if available) |
| | Log of test sediment composite preparation |
| | Sieving – size of mesh used , other sample preparation procedures/notations |
| | Holding times for all samples (test, reference, control, elutriate, lab saltwater) |

_____ _____

Signature of Laboratory Representative Date

The above signature indicates that the contracted laboratory has reviewed the data package deliverables and the data/QC summary tables and acknowledges that all of the information included in this checklist has been provided in this submittal.

# **APPENDIX H:** Checklist – Chemical Data

FORMAT AND CONTENT FOR CHEMICAL DATA PACKAGES
NYD AND EPA REGION 2 DREDGED MATERIAL PROGRAM

MATRIX (circle one): Sediment Tissue Water/Elutriate

(To be completed for each matrix)

Custody Information - Laboratories should ensure that sample custody has been maintained.

_____Custody seals or tape
_____Sample receipt form (including date and time rec'd at laboratory/cooler temperature)
_____Chain of custody record
_____Laboratory sample login report

Project Analytical Data Narrative - Discuss compliance and noncompliance with all analytical and QC requirements listed in the RTM. Provide a narrative for each of the following headings.

_____Project name
_____Parameter analyzed
_____Name of laboratory performing the analysis
_____Sample custody and processing
_____QA/QC data quality indicators/objectives
_____Analytical method summary (including any deviations, modifications or observations)
_____Holding times are specified
_____Detection limits
_____Method blanks
_____Replicate precision
_____Laboratory control sample
_____Matrix spike (MS)
_____Standard reference material (SRM)
_____Data validation (statement and signature indicating that the data was reviewed/validated)
_____Problems/corrective actions (if any)

Analytical Data Reporting

_____Names of laboratories performing the analyses
_____Name of laboratory contact
_____Table of contents
_____Raw and reduced data
_____Sampling logs/field data sheets copies of log books etc.
_____Sample tracking and traceability (from sample collection to disposal and/or archive)
_____Initial and continuing calibration checks
_____Data/QC summary tables for both wet and dry weight

A) Table Headings

_____Laboratory name (performing the analyses)
_____Project name
_____Project number
_____Analyses and medium (e.g., metals in site water and elutriate)
_____For tissue data, specify type (*M. nasuta or N. virens*)
_____Sample Identification Number (traceable to the COC documentation. Samples must be traceable from laboratory to laboratory using the same sample ID number)
_____Date sample was prepared/extracted and analyzed
_____Summary of sample weights

B) Reports results using the appropriate units.

____Metals in tissue (ug/g) wet weight and dry weight
____Metals in water (ug/L)
____Metals in sediment (ug/g) dry weight
____Organics in tissue (ng/g) wet weight and dry weight
____Organics in water (ug/L)
____Organics in sediment (ng/g) dry weight
____Dioxins/furans in tissue (pg/g) wet weight and dry weight
____Dioxins/furans in sediment (ng/g) dry weight

C) Quality Assurance and Quality Control

1) Matrix Spike Triplicate Samples Analyzed

____Sample weight/volume
____Unspiked sample results
____Spiked sample results
____Concentration of spike level used and amount of spike added
____% recovery for each analyte

2) Standard Reference Material Analyzed (if applicable)

____Sample weight/volume
____SRM contains relevant of the analytes to be tested
____Manufacturer and catalog # of SRM
____Copy of certificate of analysis (certified or consensus value)
____Results of the SRM samples
____% recovery calculated for each analyte

3) Internal/Surrogate Standard Analyzed
____Results reported for each sample analyzed
____% recovery for each internal/surrogate standard

_____ _____
Signature of Laboratory Representative Date

The above signature indicates that the contracted laboratory has reviewed the data package deliverables and the data/QC summary tables and acknowledges that all of the information included in this checklist has been provided in this submittal.