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Standard Guide for
Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing[1]

This standard is issued under the fixed designation E 1391; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers procedures for obtaining, storing, characterizing, and manipulating saltwater and freshwater sediments, for use in laboratory sediment toxicity evaluations. It is not meant to provide guidance for all aspects of sediment assessments, such as chemical analyses or monitoring, geophysical characterization, or extractable phase and fractionation analyses. However, some of this information might have applications for some of these activities. A variety of test methods are reviewed in this guide. A statement on the consensus approach then follows this review of the test methods. This consensus approach has been included in order to foster consistency among studies. The state-of-the-art is currently in its infancy, and the development of standard test methods is not feasible; however, it is crucial that there be an understanding of the significant effects that these test methods have on sediment quality evaluations. It is anticipated that recommended test methods and this guide will be updated routinely to reflect progress in our understanding of sediments and how to best study them.

1.2 There are several regulatory guidance documents concerned with sediment collection and characterization procedures that might be important for individuals performing federal or state agency-related work. Discussion of some of the principles and current thoughts on these approaches can be found in Dickson, et al **(1)**.[2]

1.3 Three documents, (Environment Canada **(2)**, USEPA **(3)** and Test Method E 1706) provide supplemental guidance on procedures dealing with the collection, storage, characterization, and manipulation of sediments used in toxicological assessments.

1.4 This guide is arranged as follows:

1.5 Field-collected sediments might contain potentially toxic materials and should thus be treated with caution to minimize occupational exposure to workers. Worker safety must also be considered when working with spiked sediments containing various organic, inorganic, or radiolabeled contaminants, or some combination thereof. Careful consideration should be given to those chemicals that might biodegrade, volatilize, oxidize, or photolyze during the exposure.

1.6 The values stated in either SI or inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[3]

D 4387 Classification of Grab Sampling Devices for Collecting Benthic Macroinvertebrates[4]

D 4822 Guide for Selection of Methods of Particle Size Analysis of Fluvial Sediments (Manual Methods)[5]

D 4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments[5]

E 729 Guide for Conducting Acute Tests with Fishes, Macroinvertebrates, and Amphibians[4]

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[1] This guide is under the jurisdiction of ASTM Committee E47 on Biological Effects and Environmental Fate and is the direct responsibility of Subcommittee E47.03 on Sediment Toxicology.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.05.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

E 943 Terminology Relating to Biological Effects and Environmental Fate[4]

E 1367 Guide for Conducting 10-Day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods[4]

E 1383 Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates[4]

E 1706 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates[3]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[5]

3. Terminology

3.1 *Definitions:*

3.1.1 The words "must," "should," "may,"" can," and "might" have very specific meanings in this guide. "Must" is used to express an absolute requirement, that is, to state that the test ought to be designed to satisfy the specified condition, unless the purpose of the test requires a different design. "Must" is used only in connection with the factors that relate directly to the acceptability of the test. "Should" is used to state that the specified condition is recommended and ought to be met in most tests. Although the violation of one "should" is rarely a serious matter, the violation of several will often render the results questionable. Terms such as "is desirable,"" is often desirable," and" might be desirable" are used in connection with less important factors. "May" is used to mean "is (are) allowed to," "can" is used to mean" is (are) able to," and "might" is used to mean "could possibly." Thus, the classic distinction between "may" and" can" is preserved, and "might" is never used as a synonym for either "may" or "can."

3.1.2 For definitions of terms used in this guide, refer to Guide E 729, Terminologies D 1129 and E 943, and Classification D 4387; for an explanation of units and symbols, refer to IEEE/ASTM SI 10.

4. Summary to Guide

4.1 This guide provides a review of widely used test methods for collecting, storing, characterizing, and manipulating sediments for toxicity testing. Where the science permits, recommendations are provided on which procedures are appropriate, while identifying their limitations.

5. Significance and Use

5.1 Sediment toxicity evaluations are a critical component of environmental quality and ecosystem impact assessments, used to meet a variety of research and regulatory objectives. The manner in which the sediments are collected, stored, characterized, and manipulated can influence the results of any sediment quality or process evaluation greatly. Addressing these variables in a systematic and uniform manner will aid the interpretations of sediment toxicity or bioaccumulation results and may allow comparisons between studies.

6. Interferences

6.1 Maintaining the integrity of a sediment sample relative to ambient environmental conditions during its removal, transport, and testing in the laboratory is extremely difficult. The sediment environment is composed of a myriad of microenvironments, redox gradients, and other interacting physicochemical and biological processes. Many of these characteristics influence sediment toxicity and bioavailability to benthic and planktonic organisms, microbial degradation, and chemical sorption. Any disruption of this environment complicates interpretations of treatment effects, causative factors, and in situ comparisons. See Section 9 for additional information.

7. Apparatus

7.1 A variety of sampling, characterization, and manipulation methods exist using different equipment. These are reviewed in Sections 9 and 14.

7.2 *Cleaning*—Test chambers and equipment used to collect and store sediment samples, prepare and store dilution water and stock solutions, and expose test organisms should be cleaned before use. New glassware and plasticware should be soaked in 1:1 concentrated acid prior to use. Soaking overnight is adequate for glassware. Soaking for seven days in HCl, followed by seven days in HNO_3, followed by seven days in deionized water is recommended for plasticware. Used sample containers should be washed following these steps: (*1*) nonphosphate detergent wash, (*2*) triple water rinse, (*3*) water-miscible organic solvent wash (acetone followed by pesticide-grade hexane **(4, 5)**, (*4*) water rinse, (*5*) acid wash (such as 5 % concentrated hydrochloric acid), and (*6*) triple rinse with deionized-distilled water. Altering this cleaning procedure might result in problems. Many organic solvents might leave a film that is insoluble in water (Step 3). A dichromate-sulfuric acid cleaning solution can generally be used in place of both the organic solvent and the acid (Steps 3 through 5), but it might attack silicone adhesive. (See 9.10 for cleaning during sample collection.)

8. Safety Hazards

8.1 Many substances can affect humans adversely if adequate precautions are not taken. Information on the toxicity to humans **(6)** and recommended handling procedures of toxicants **(7)** should be studied before tests are begun with any contaminant or sediment. Health and safety precautions should be incorporated into any study plan prior to initiating any work with contaminants or sediments.

8.2 Field-collected sediments might contain a mixture of hazardous contaminants or disease-causing agents such that proper handling to avoid human exposure is critical. Skin contact with all test materials and solutions should therefore be minimized by such means as wearing appropriate protective gloves, especially when putting hands into sediments, overlying water, or washing equipment. Proper handling procedures might include the following: (*1*) sieving and distributing sediments under a ventilated hood or enclosed glove box; (*2*) enclosing and ventilating the toxicity test water bath; and (*3*) using respirators, aprons, safety glasses, and gloves when handling potentially hazardous sediments. Special procedures might be necessary with radiolabeled test materials **(8)** and materials that are, or are suspected of being, carcinogenic **(7)**.

8.3 The disposal of sediments, dilution water over sediments, and test organisms containing hazardous compounds might pose special problems. Removal or degradation of the toxicant(s) before disposal is sometimes desirable for tests involving spiking sediments with known toxicants. Disposal of all hazardous wastes should adhere to the requirements and regulations of the Resource Conservation and Recovery Act and any relevant state or local regulations.

9. Sampling and Transport

9.1 Sediments have been collected for a variety of chemical, physical, toxicological, and biological investigations. The sediments should be collected from depositional zones in which fine-grained sediments accumulate. Site selection should also consider the location of pollutant loadings and hydrological flow patterns. The site selection may also need to be of a random or stratified random nature, depending on the study objectives. Sediment variability must be considered since most sediments are very heterogeneous (both vertically and horizontally) in nature. A preliminary survey or review of background data may therefore be required to determine accurately the appropriate number of sediment replicates to collect.

9.2 Sediment collections have been made with grab and dredge sampling devices and core samplers (see Table 1 and Guide D 4823). The advantages and disadvantages of the various collection methods have been reported previously **(9, 10)** and are summarized in Table 2. All sampling methods disturb the sediment integrity to a degree. It is important to obtain sediments with as little disruption as possible when using sediment toxicity evaluations for realistic laboratory evaluations of in situ conditions. Core sampling is preferred above other methods for this reason. Choosing the most appropriate sediment sampler for a study will depend on the sediment's characteristics, efficiency required, and study objectives. Several references are available that discuss the various collection devices **(9-13)**. Grab samplers can penetrate sediments to depths of 10 to 50 cm. Dredge samplers collect to a depth of 10 cm and disrupt sediment integrity. Core samplers collect up to 1 or 2 m when collected by hand or gravity. However, vibratory or piston corers can reach depths of 10 m. The depth of penetration is limited to 10 core diameters in sandy substrates and 20 diameters in predominately clay sediments. The efficiency of these samplers for benthic collections has been compared, and the grab samplers are less efficient collectors than the corers in general, but they are easier to handle in rough water, often require fewer personnel, and are obtained more easily **(11, 13, 14)**. Most of the reported studies used grab samplers, although box corers **(15-17)**, gravity corers **(18)**, and hand collection **(19-21)** test methods are reported with increasing frequency.

9.3 The disadvantages of grab and dredge samplers (Table 2) include a shallow depth of penetration and the presence of a shock wave that results in loss of the fine surface sediments. Murray and Murray **(22)**, however, described a grab sampler

TABLE 1 Sampling Containers, Preservation Requirements, and Holding Times for Sediment Samples[A] (EPA, 196, 197). See also Rochon and Chevalier (160)

Contaminant	Container[B]	Preservation	Holding Time
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days
Sulfide	P, G	Cool, 4°C	28 days
Sulfite	P, G	Cool, 4°C	48 h
Nitrate	P, G	Cool, 4°C	48 h
Nitrate-nitrite	P, G	Cool, 4°C	28 days
Nitrite	P, G	Cool, 4°C	48 h
Oil and grease	G	Cool, 4°C	28 days
Organic	P, G	Cool, 4°C	28 days
Metals[C]			
Chromium VI	P, G	Cool, 4°C	48 h
Mercury	P, G		8 days
Metals (except Cr or Hg)	P, G		6 months
Organic Compounds[C]			
Extractables (including phthalates, atrosamines organochlorine pesticides, PCB's artroaromatics, isophorone, Polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons, and TCDD)	G, PTFE-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Extractractables (phenols)	G, PTFE-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Purgables (halocarbons and aromatics)	G, PTFE-lined septum	Cool, 4°C	14 days
Purgables (acrolein and acrylonitrate)	G, PTFE-lined septum	Cool, 4°C	3 days
Orthophosphate	P, G	Cool, 4°C	48 h
Pesticides	G, PTFE-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)
Phenols	P, G	Cool, 4°C	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 h
Phosphorus, total	P, G	Cool, 4°C	28 days
Chlorinated organic compounds	G, PTFE-lined cap	Cool, 4°C	7 days (until extraction) 30 days (after extraction)

[A] Taken from EPA 600-4-84-075 and EPA 600-4-85-048. See also Ref **(85)** and USEPA/COE 1991.

[B] Polyethylene (P) or Glass (G).

[C] Freezing is recommended by some for metals and organics with holding times of 30 and 10 days, respectively (USEPA/COE 1991).

TABLE 2 Summary of Bottom Sampling Equipment[A]

Device	Use	Advantages	Disadvantages
PTFE or glass tube	Shallow wadeable waters or deep waters if SCUBA available. Soft or semi-consolidated deposits.	Preserves layering and permits historical study of sediment deposition. Rapid—Samples immediately ready for laboratory shipment. Minimal risk of contamination.	Small sample size requires repetitive sampling.
Hand corer with removable PTFE or glass liners	Same as above except more consolidated sediments can be obtained.	Handles provide for greater ease of substrate penetration. Above advantages.	Careful handling necessary to prevent spillage. Requires removal of liners before repetitive sampling. Slight risk of metal contamination from barrel and core cutter.
Box corer	Same as above.	Collection of large sample undisturbed, allowing for subsampling.	Hard to handle.
Gravity corers, that is, Phleger Corer	Deep lakes and rivers. Semi-consolidated sediments.	Low risk of sample contamination. Maintains sediment integrity relatively well.	Careful handling necessary to avoid sediment spillage. Small sample, requires repetitive operation and removal of liners. Time consuming.
Young Grab (PTFE- or kynar-lined modified 0.1-m^2 van Veen)	Lakes and marine areas.	Eliminates metal contamination. Reduced bow wake.	Expensive. Requires winch.
Ekman or box dredge	Soft to semisoft sediments. Can be used from boat, bridge, or pier in waters of various depths.	Obtains a larger sample than coring tubes. Can be subsampled through box lid.	Possible incomplete jaw closure and sample loss. Possible shock wave, which may disturb the fines. Metal construction may introduce contaminants. Possible loss of "fines" on retrieval.
PONAR Grab Sampler	Deep lakes, rivers, and estuaries. Useful on sand, silt, or clay.	Most universal grab sampler. Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb" fines." Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-53 Piston Corer	Waters of 4 to 6 ft deep when used with extension rod. Soft to semi-consolidated deposits.	Piston provides for greater sample retention.	Cores must be extruded on site to other containers. Metal barrels introduce risk of metal contamination.
Van Veen	Deep lakes, rivers, and estuaries. Useful on sand, silt, or clay.	Adequate on most substrates. Large sample obtained intact, permitting subsampling.	Shock wave from descent may disturb "fines." Possible incomplete closure of jaws results in sample loss. Possible contamination from metal frame construction. Sample must be further prepared for analysis.
BMH-60	Sampling moving waters from a fixed platform.	Streamlined configuration allows sampling where other devices could not achieve proper orientation.	Possible contamination from metal construction. Subsampling difficult. Not effective for sampling fine sediments.
Petersen Grab Sampler	Deep lakes, rivers, and estuaries. Useful on most substrates.	Large sample; can penetrate most substrates.	Heavy. May require winch. No cover lid to permit subsampling. All other disadvantages of Ekman and Ponar.
Shipek Grab Sampler	Used primarily in marine waters and large inland lakes and reservoirs.	Sample bucket may be opened to permit subsampling. Retains fine- grained sediments effectively.	Possible contamination from metal construction. Heavy. May require winch.
Orange-Peel Grab Smith-McIntyre Grab	Deep lakes, rivers, and estuaries. Useful on most substrates.	Designed for sampling hard substrates.	Loss of fines. Heavy. May require winch. Possible metal contamination.
Scoops, Drag Buckets	Various environments, depending on depth and substrate.	Inexpensive, easy to handle.	Loss of fines on retrieval through water column.

[A] Comments represent subjective evaluations.

usable in rough water that samples the top 1 cm of sediment quantitatively and retains fine materials. Other grab samplers that sample surface sediments quantitatively have been described by Grizzle **(23)**. The depth profile of the sample may be lost in removal of the sample from the sampler. Grab sampling promotes the loss of not only fine sediments (Table 2), but also water-soluble compounds and volatile organic compounds present in the sediment. Dredge samplers are appropriate only for collecting sediments that are to be dredged because they disrupt sediment integrity severely and lose surficial fines.

9.4 Studies of macroinvertebrate sampling efficiency with various grab samplers have provided useful information for sampling in sediment toxicity and sediment quality evaluations. These data provide information that would indicate sampler efficiency at retaining surficial sediment layers. The modified van Veen is used commonly in coastal sampling **(24)**. The Ekman grab is a commonly used sampler for benthic investigations **(23)**. The Ekman's efficiency is limited to less compacted, fine-grained sediments, as are the corer samplers. Blomqvist **(25)** reviewed the various Ekman modifications and their associated problems and concluded that the Ekman grab could be used reliably if caution was used during operation. The most commonly used corer is the Kajak-Brinkhurst corer. The Petersen, PONAR, and Smith-McIntyre grabs are used most often **(11)** in more resistant sediments. Based on studies of benthic macroinvertebrate populations the sediment corers are the most accurate samplers, in most cases followed by the Ekman grab **(11)**. The PONAR grab was the most accurate and the Petersen the least for compacted sediments **(11)**. A comparison of sampler precision indicated the van Veen sampler to be the least precise; the most precise were the corers and Ekman grab **(11).**

9.5 Many of the problems associated with grab and dredge samplers are largely overcome with the corers. The best corers for most sediment studies are hand-held poly tetrafluoroethylene (PTFE) plastic, high-density polyethylene, glass corers (liners), or large box-corers. The corers can maintain the integrity of the sediment surface while collecting a sufficient depth. Furthermore, the box core can be sub-cored or sectioned at specific depth intervals, as required by the study. Unfortunately, the box corer is large and cumbersome; it is thus difficult to use. Freefall or gravity cores tend to cause compaction, disrupting the vertical gradients in the sediment. Compaction is reduced using the piston corer. Other coring devices that have been used successfully include the percussion corer **(26)** and vibratory corers **(27-29)**.

9.6 Corer samplers have several limitations. Most corers do not work well in sandy sediments; grab samplers or diver-collected material remain the only current alternatives. In general, corers collect less sediment than grab samplers, which may provide inadequate quantities for some studies. Small cores tend to increase bow waves (that is, disturbance of surface sediments) and compaction, thus altering the vertical profile. However, these corers provide better information on spatial variation when multiple cores are obtained **(11, 30-34)**. As shown by Rutledge and Fleeger **(35)** and others, care must be taken in subsampling from core samples since surface sediments might be disrupted in even hand-held core collection. They recommend subsampling in situ or homogenizing core sections before subsampling. See Ref **(10)** for additional information of various core types.

9.7 Core sampling should be used to best maintain the complex integrity of the sediment for studies of sediment toxicity, interstitial waters, microbiological processes, or chemical fate. When obtaining cores from shallow waters, one must ensure that the vessel does not disturb the sediments before sampling **(36)**. If core sampling is not possible due to an inability of the core to penetrate the sediment (for example, highly compacted sediment) or retain the sample (for example, primarily sand composition), grab samplers should be used that reduce the loss of fine-grained surficial sediments.

9.8 Subsampling, compositing, or homogenization of sediment samples is often necessary, and the optimal methods will depend on the study objectives. Important considerations include the following: loss of sediment integrity and depth profile; changes in chemical speciation by means of oxidation and reduction or other chemical interactions; chemical equilibrium disruption resulting in volatilization, sorption, or desorption; changes in biological activity; completeness of mixing; and sampling container contamination. It is advantageous in most studies of sediment toxicity to subsample the inner core area (not contacting the sampler) since this area is most likely to have maintained its integrity and depth profile and not be contaminated by the sampler. Subsamples from the depositional layer of concern, for example, the top 1 or 2 cm, should be collected with a nonreactive sampling tool such as a PTFE-lined calibration scoop **(37)**. Samples are frequently of a mixed depth; however, a 2-cm sample **(38)** is the most common depth obtained, although depths up to 12 m have been used in some dredging studies. It is advantageous or necessary for some studies to composite or mix single sediment samples **(37, 39, 40)**. Composites usually consist of three to five grab samples. An advantage of composited samples is that they reduce the likelihood of missing a "hot spot" due to site heterogeneity. However, a disadvantage is the loss of information on the spatial variability at the site and reduction of the toxicity of hot spot samples when they are diluted with cleaner samples. This is a more critical issue in the boundary areas of the site contamination, also known as the grey zone. Subsamples are collected with a nonreactive sampling scoop and placed in a nonreactive bowl or pan. The composite sample should be mixed until the texture and color appear uniform.

9.9 The assessment of in situ sediment toxicity or bioaccumulation is aided by the collection and testing of reference and control samples. For the purposes of this guide, a reference sediment is defined as a whole sediment near an area of contamination used to assess sediment condition exclusive of the material(s) of interest. It should contain characteristics similar to those of the test sediment. Sediment characteristics, such as particle size distribution and percent organic carbon, should bracket that of the test sediment. If a wide range of test sediment types exists, the reference sediment characteristics should be in an intermediate range unless the test species is affected by particle size. The appropriate ASTM guides for marine (Guide E 1367) and freshwater (Guide E 1383) invertebrates should then be consulted to determine the particle size requirements of the test species. It is preferable that reference sediments be collected from the same aquatic system and be located close to and have physical, chemical, and biological characteristics similar to those of the test sediment. The reference sediment test results might be analyzed as either a treatment or a control variable, depending on the study objectives. The reference sediment might be toxic in some situations due to naturally occurring chemical, physical, or biological properties. It is important for this reason to also use control sediments in the evaluation of test sediments. A control sediment is defined as a sediment that is essentially free of contamination and is used routinely to assess the acceptability of a test. Control sediments have been used successfully in toxicity evaluations **(41)**.

9.10 When collecting sediment grab samples, it is important to clean the sampling device, scoop, spatula, and mixing bowls between sample sites. The cleaning procedure can follow that outlined in Section 7 or the following **(42)**: (*1*) soap and water wash, (*2*) distilled water rinse, (*3*) methanol rinse, (*4*) methylene chloride rinse, and (*5*) site water rinse. Waste solvents should be collected in labeled hazardous waste containers.

9.11 The transport conditions for the samples were not specified in the references reviewed in most cases. Where conditions were specified, the sediments were usually transported whole, in both plastic, polyethylene **(43-45)**, and glass **(20, 21, 46)** containers, and transported under refrigeration or on ice **(20, 21, 38, 46-51)**.

9.12 The collection, transport, storage, and test chamber material composition should be chosen based on a consideration of sorption effects, sample composition, and contact time (Table 1). For example, in sediments in which organics are of concern, brown borosilicate glass containers with PTFE lid liners are optimal, while plastic containers are recommended for metal samples. PTFE or high-density polyethylene containers are relatively inert and optimal for samples contaminated with multiple chemical types. Additionally, polycarbonate containers have been shown not to sorb metal species **(52)**. However, Moody and Lindstrom **(53)** found that all plastics (including PTFE) leached elements and should be preconditioned with a seven-day soaking in 1:1 HCl, HNO_3, and deionized water. Shipping containers with insulation 1 in. (25.4 mm) in thickness kept samples at 4°C for 21 h, while insulation of 2-in. (51-mm) thickness maintained 4°C for 60 to 82 h **(54)**. Additional information regarding chemical analyses on sample containers, preservation, storage times, and volume requirements is available in other guidance documents **(9, 10, 39, 55-57)**. These criteria are applicable to toxicity test requirements in many cases.

10. Storage

10.1 Containers for storage were generally not specified, although it was assumed that the containers were the same as the transport containers, where specified, and were generally high-density polyethylene (see 9.12). Where sediments contain volatile compounds, transport and storage should be in airtight PTFE or glass containers with PTFE-lined screw caps. Volatile and semi-volatile compounds must be stored at 4°C and are lost in seven or eight days, respectively **(54)**. See Table 1 for further information on the storage requirements for chemical analyses.

10.2 Drying, freezing, and cold storage conditions all affect toxicity and bioavailability **(58-63)**. The storage time of sediments used in toxicity tests was often not specified and, where specified, ranged from a few days **(64)** to one year **(44)**. The storage of sediments after arrival at the laboratory was generally by refrigeration at 4°C **(43-45, 47-51, 61, 64-67)**. Significant changes in metal toxicity to cladocerans and microbial activity have been observed in stored sediments **(62, 68)**. Recommended limits for the storage of metal-spiked sediments have ranged from within two days **(58)** to five days **(64)** and seven days **(69, 70)**. Cadmium toxicity in sediments has been shown to be related to acid volatile sulfide (AVS) complexation **(71)**. When anoxic sediments were exposed to air, AVS was volatilized rapidly. AVS is apparently the reactive solid phase sulfide pool that binds metal, thus reducing toxicity. If a study objective is to investigate metal toxicity and the sediment environment is anoxic, exposure to air might reduce or increase toxicity due to the oxidation and precipitation of the metal species or loss of acid volatile sulfide complexation. A study of sediments contaminated with nonpolar organics found that the interstitial water storage time did not affect toxicity to polychaetes when samples were frozen **(72)**; however, it is generally agreed that sediments to be used for toxicity testing must not be frozen **(61, 63, 64, 69, 73)**.

10.3 Although risking changes in sediment composition, several studies elected to freeze samples **(38, 61, 74-78)**. Fast-freezing of sediment cores has been recommended for some metal and organic chemical analyses **(24, 65, 79)**; however, this alters the sediment structure and profile distortion occurs **(35)**. Freezing has been reported to inhibit the oxidation of reduced iron and manganese compounds **(75)**. It has also been recommended for stored sediments that are to be analyzed for organics and nutrients **(80)**. Thomson, et al **(79)** found that no storage method for sediments preserved the initial chemical and physical characteristics of the sediment. Freezing was adequate for the chemical analyses of several metals and organic material **(65, 79)**. Changes were observed at 15 days in sediments stored at 4°C. Oxidation was greater than reduction during storage **(79)**. Carr and Wilkniss **(81)** showed no mercury loss in sediments acidified to pH 1 for up to eight days, but the sorbed fraction decreased from 80 to 15 % of the total concentration. If sediments are to be frozen for chemical analyses, they should be a split sample from those used for toxicity testing that are kept at 4°C.

10.4 Interstitial water chemistry can change significantly after 24-h storage **(82, 83)**, even when stored at in situ temperatures **(83)**. The coagulation and precipitation of humic material was noted when interstitial water was stored at 4°C for more than one week **(84)**. Oxidation of reduced arsenic species in the pore water of stored sediments was unaffected for up to six weeks when samples were acidified and kept near 0°C, without deoxygenation. Deoxygenation was necessary when samples were not acidified **(85)**. See also Section 11.

10.5 In summary, it is recommended that sediments for toxicity tests and chemical analyses be refrigerated or placed on ice in polyethylene containers during transport. In addition, if samples are to be used for chemical analyses, the appropriate container and holding time should be used as previously described and in Table 1. The storage conditions should be refrigeration at 4°C and under anoxic conditions, if appropriate **(39, 55, 86)**. It has been shown that some contaminated sediments can be stored at 4°C for up to 12 months without significant alterations in toxicity **(87)**. Limits to storage time before testing therefore appear to be a function of both the sediment and contaminant characteristics. Storage should be limited to a two-week period at 4°C unless previous data exist that indicate that the study site sediments can be stored without affecting toxicity.

11. Collection of Interstitial Water

11.1 Interstitial water (pore water), defined as the water occupying the space between sediment or soil particles, is often isolated to provide either a matrix for toxicity testing or an indication of the concentration and partitioning of contaminants within the sediment matrix. There is some indication that the interstitial water may be as useful as whole sediment for evaluating the toxicity of some sediment-associated compounds, for example, those that are not sorbed strongly to particles and where the ingestion of contaminated particles is not a major route of accumulation. The isolation of sediment interstitial water can be accomplished by several methods: centrifugation, squeezing, gas pressurization, suction, and equilibrium dialysis. These techniques have been reviewed recently by Adams **(88)** and Burton **(89)**. In general, where relatively large volumes of water are required only centrifugation (for example, **46, 84, 90-98)** and sediment squeezing **(72, 99)** can provide large quantities. Other methods, such as suction **(100-103)** and in situ samplers **(88)**, do not produce sufficient volumes from most sediments easily.

11.2 Most collection methods have been shown to alter interstitial water chemistry and therefore may alter toxicity. There are a number of precautions that one should take to reduce the likelihood of causing significant sample change from in situ conditions. Some interstitial water constituents, for example, dissolved organic carbon, dimethylsulfide, ammonia, and major cations, can be altered significantly by the collection method **(104-107)**. Increased sample handling by means of methods such as centrifugation or squeezing, compared to in situ "peepers" or core-port suction, may cause increased ammonia and decreased sulfide concentrations **(105)**. Other constituents, such as salinity, dissolved inorganic carbon, ammonia, sulfide, and sulfate, might not be affected by collection, providing that oxidation is prevented **(105)**. If the sediments are anoxic, all of the steps involved in sample processing should be conducted in inert atmospheres or by limited contact with the atmosphere in order to prevent oxidation (and the subsequent sorption and precipitation) of reduced species **(104, 105, 108)**. Immediate collection of the interstitial water is recommended since chemical changes might occur even when the sediments are stored for short periods of time (for example, 24 h) at in situ temperatures **(82, 83)**. Toxicity changes have been observed in interstitial water stored for less than 24 h **(109)**. The coagulation and precipitation of humic material was noted when interstitial water was stored at 4°C for more than one week **(84)**. The oxidation of reduced arsenic species in the interstitial water of stored sediments was unaffected for up to six weeks when the samples were acidified and kept near 0°C, without deoxygenation. Deoxygenation was necessary when the samples were not acidified, **(85)**. Others have recommended that interstitial waters be frozen after extraction, prior to toxicity testing, to prevent changes **(72)**. The optimal collection method will depend on the intended use of the sample (for example, acidification for metal analysis and not toxicity testing), characteristics of the sediment, and contaminants of concern.

11.3 The conditions for isolation of interstitial waters by centrifugation have varied considerably. Interstitial waters have been isolated for toxicity testing over a range of centrifugal forces and temperatures **(46, 84, 90-95)** with centrifuge bottles of various compositions. When centrifugation followed by filtration has been compared with in situ dialysis, higher speed centrifugation followed by filtration with 0.2 membrane filters has produced results that were more comparable for metals and organic carbon **(75, 109, 110)**. Centrifuging at low speeds or the use of a 0.45-µm pore size membrane will result in the collection of both dissolved contaminants, colloidal materials, and aquatic bacteria **(75)**. High-speed centrifugation (for example, 10 000 × g) is necessary to remove colloids and dispersible clays **(88, 111, 112)**. The duration of the centrifugation has varied in the literature, but 30 min is relatively common and is the recommended time. The temperature for the centrifugation should reflect the ambient temperature of collection to ensure that the equilibrium between the particles and interstitial water is not shifted. Since trace metals and organics concentrate on solids, their removal is important in sorption and partitioning studies **(75, 111)**. However, filtration through a wide range of filter types such as glass fiber or polycarbonate membranes may be inappropriate since they sorb some dissolved metals and organics **(113)**. If filtration is used, a nonfiltered sample should also be tested for toxicity and contaminant concentrations. The effects of centrifugation speed, filtration, and oxic conditions on some chemical concentrations in interstitial waters have been well documented (for example, **88, 114, 115**). It is recommended that sediments should be centrifuged at 10 000 × g for a 30-min period for routine toxicity testing of interstitial waters.

11.4 It is difficult to collect interstitial water from sediments that are predominately coarse sand. A modified centrifuge bottle has been developed, with an internal filter that can recover 75 % of the interstitial water, compared to 25 to 30 % from squeezing **(116)**.

11.5 If sorptive organic compounds or mixtures of inorganic and organic compounds are to be isolated, PTFE centrifuge bottles should be used. Polytetrafluorethylene bottles will collapse at 3000 × g but have been used successfully in the range of 2500 g when filled to 80 % of capacity **(90)**. So, in this case, the isolation of interstitial water should be at the temperature of collection, at a slower speed of 2500 g for 30-min duration. This material will contain colloidal material as well as dissolved compounds. Removal of the colloids may not be possible at low centrifugation speeds, without filtration. The influence of dissolved and colloidal organic carbon may be estimated by measuring the organic carbon content. Centrifugation can be performed with glass tubes (up to 10 000 × g) **(113)** if small volumes of water are required, for example, 50 mL, for testing higher speed. High-speed centrifugation in stainless steel centrifuge tubes can be performed if metals are not an issue.

11.6 The isolation of interstitial water by squeezing has been performed by means of a variety of practices **(72, 88, 96-99, 117)**. In all cases, the interstitial water is passed through a filter that is a part of the apparatus. Filters have different sorptive capacities for different compounds. The characteristics of filters and the filtering apparatus should be considered carefully based on the types of contaminants expected. Squeezing has been demonstrated to yield results equivalent to those for other methods for silica **(118)** but not for sulfide **(119)**. However, squeezing has been shown to produce a number of artifacts due to shifts in equilibrium from pressure, temperature, and gradient changes (for example, **97, 106, 107, 120-122**). Squeezing can affect the electrolyte concentration in the interstitial water with a drop near the end of the squeezing process. It is therefore recommended that moderate pressures be used with electrolyte (conductivity) monitoring during extraction **(121)**. Several studies revealed significant alterations to the interstitial water composition when squeezing was at temperatures that differed from ambient temperatures (for example, **106, 107**). The major sources of alteration of the interstitial water, when using the squeezing method, are as follows: contamination from overlying water, internal mixing of the interstitial water during extrusion, and solid-solution reactions as the interstitial water is expressed through the overlying sediment. As interstitial waters are displaced into upper sediment zones during squeezing, they come into contact with solids that they are not in equilibrium with. This intermixing causes solid-solution reactions to occur. These reactions will generally reflect an approach to saturation, adsorption or desorption, and ion exchange. The chemistry of the sample may be altered due to the fast kinetics (minutes to hours) of these reactions. Most interstitial water species are out of metastable equilibrium with overlying sediments and are transformed rapidly, such as the case observed with ammonia and trace metals **(123, 124)**. Bollinger, et al **(120)** found elevated levels of several ions and dissolved organic carbon in squeezed samples compared to samples collected by peepers. The degree of artifact will depend on the element, sediment characteristics, and redox potential. It is unlikely that reactive species gradients can be established by means of the squeezing of sediment cores **(118)**.

11.7 Small-volume isolation of interstitial water, generally for chemical analysis, can also be performed by vacuum filtration **(75, 103, 125)**, gas pressurization **(37, 98)**, or displacement after removing the sediment from the aquatic environment **(88)**. When preparing the sediments for interstitial water isolation of metals, care must be taken to maintain the anoxic conditions of deeper sediments by performing the procedures under an inert atmosphere **(88)**. When core suction was compared to centrifugation and squeezing, it was found that the recovery of spiked tritium was similar; however, chlorobenzene differed significantly among methods, with suction exhibiting the highest recovery, followed by squeezing and centrifugation **(103)**. Suction using an aquarium air stone recovered up to 1500 mL from sediment (4 L) and suctioned in an anoxic environment **(124)**. Problems common to suction methods are a loss of equilibration between the interstitial water and the solids, filter clogging, and oxidation **(126)**. However, in situ suction or suction by means of core ports has been shown to define small gradients of some sediment-associate compounds accurately, including ammonia, which can change an order of magnitude over a 1-cm depth **(115)**. However, these definitive suction methods do not provide an adequate volume for conducting most toxicity test procedures.

11.8 Perhaps the optimal method of pore water collection is by the use of equilibrium dialysis **(88, 127-130)** or in situ suction techniques **(100-103, 105)**. These methods have the greatest likelihood of maintaining in situ conditions and have been used to sample dissolved gases **(131)** and volatile organic compounds **(103)**. However, these techniques isolate only relatively small volumes of interstitial water and must be placed by divers in deeper waters which limits the depth and conditions at which the devices can be deployed. Suction of undisturbed sediments is also possible from intact box core-collected sediments. The duration of equilibration for dialysis has ranged from hours to a month, but one to two weeks is most often used **(88)**. The optimal equilibration time is a function of the sediment type, contaminants of concern, and temperature (for example, **105, 115, 129, 132-134**). Many of the artifact problems associated with dialysis samplers have been discussed **(110)**. The total organic carbon may be elevated in peepers (4 to 8-µm pore size) due to biogenic production; however, colloidal concentrations are lower than in centrifuged samples **(111)**. When ionizable compounds, for example, metals, are to be collected, it is important to pre-equilibrate the samplers with an inert atmosphere in order to avoid introducing oxygen into the sediments and thereby changing the equilibrium. Plastic samplers can contaminate anoxic sediments with diffusable oxygen and should be stored before testing in inert atmospheres **(132)**. In addition, samples should also be kept under an inert atmosphere and processed quickly when they are collected and processed. Cellulose membranes are unsuitable because they decompose too quickly. A variety of polymer materials have been used, some of which may be inappropriate for studies of certain nonpolar compounds. However, efforts to use semipermeable membrane devices filled with a nonpolar sorbant show some promise for use in dialysis systems for organic compounds **(135)**. Test organisms have recently been exposed within peeper chambers in which larger mesh sizes of 149 µm were used successfully in oxic sediments **(134, 136, 137)**. Equilibration of conductivity was observed within hours of peeper insertion into the sediment. Replicate peepers revealed extreme heterogeneity in sediment interstitial water concentrations of ammonia and dissolved oxygen. Sediments that were high in clay and silt fractions were usually anoxic and did not allow for organism exposure in situ.

11.9 Based on the literature previously discussed, no clear superior method exists for isolating interstitial water for toxicity testing purposes. Each approach has unique strengths and weaknesses that vary with the sediment's characteristics, contaminants of concern, toxicity test methods to be used, and resolution necessary (that is, the data quality objectives). For most toxicity test procedures, relatively large volumes of interstitial water (for example, litres) are frequently needed for static or static renewal exposures with the associated water chemistry analyses. The use of in situ methods are preferred if smaller volumes are adequate and logistics allow because they are less likely to produce sample artifacts. The collection of core samples that are then subjected to immediate side port suctioning or centrifugation at ambient bottom water temperatures is recommended if logistics do not permit the placement of in situ samplers. However, it will be necessary for most studies to collect larger quantities of samples, preferably multiple cores, that are processed in an inert environment and centrifuged at ambient temperatures as rapidly as possible. If other methods and procedures are used for interstitial water collection (such as grab samplers, exposure to oxygen, extraction at room temperature, delayed extraction, squeezing, and filtration), the investigator should realize that the interstitial water sample has been altered from in situ conditions.

12. Characterization

12.1 Sediments that are to be analyzed for toxicity should be characterized physically and chemically. At a minimum, this characterization should include moisture content (total solids and specific gravity), organic carbon or volatile matter content, and particle size. More extensive characterization may be necessary to meet the study objectives. The degree of precision and accuracy necessary for these analyses will depend on the study's data quality objectives. By their nature, sediments are very heterogenous; they exhibit significant temporal and spatial heterogeneity in the laboratory and in situ. Lappalainen **(138)** demonstrated seasonal effects on interstitial water chemistry due to differences between sediment and overlying water temperature. Convectional heat transfer, interstitial water currents, and the transfer of soluble and gaseous materials was observed in the spring and autumn to sediment depths of tens of centimetres. Replicate samples should be analyzed to determine the variance in sediment characteristics and analytical methods. Sediment characterization will depend on the study objectives and contaminants of concern. Several additional characteristics that may assist in data interpretation and the quality assurance (QA)/quality control (QC) process (that is, assessing sediment integrity, artifact production, optimal extraction, and test procedures) include the following: in situ temperature, ash-free weight (total volatile solids), total and dissolved organic carbon (determined by titration or combustion), pore water salinity (for estuarine and marine sediments), pH, Eh, ammonia, and cation-exchange capacity. Many of the characterization methods have been based on analytical techniques for soils and waters, and the literature should be consulted for further information **(13, 139-141)**.

12.2 The moisture content of sediments is measured by drying the sediments at 50 to 105°C to a consistent weight **(13)**.

12.3 Volatile matter content is often measured instead of, and in some cases in addition to, organic carbon content as a measure of the total amount of organic matter in a sample. This measurement is made by ashing the sediments at high temperature and reporting the percent ash-free dry weight **(142-144)**. Although the exact method for ashing the sample is often not specified, the normally accepted temperature is 550± 50°C **(13, 39)** for 2 to 24 h.

12.4 Carbon fractions that may be of importance in determining toxicant fate and bioavailability include the following: total organic carbon **(39, 145-147)**, dissolved organic carbon **(84)**, dissolved inorganic carbon, sediment carbonates, and reactive particulate carbon **(148, 149)**. Reactive particulate carbon is that portion which equilibrates with the aqueous phase. Sediment organic carbon content has been measured by wet oxidation, which is also useful for determination of the organic carbon content of water **(150)**. Organic carbon analyses have also been conducted by titration **(151)**, modification of the titration method **(152)**, or combustion after the removal of carbonate by the addition of HCl and subsequent drying **(67)**.

12.5 Sediment particle size can be measured by numerous methods **(139, 153**, and see Guide D 4822), depending on the particle properties of the sample **(154)**. Greater agreement exists between sizing and settling methods when the clay fractions are greater than 15 % **(155)**. Particle size distribution is often determined by wet sieving **(4, 13, 39, 139, 155)**. Particle size classes might also be determined by the hydrometer method **(156, 157)**, pipet method **(139, 158)**, settling techniques **(159)**, X-ray absorption **(155, 158)**, and laser light scattering **160)**. The pipet method may be superior to the hydrometer method **(161)**. A method using a Coulter (particle size) counter might be used **(162, 163)** to obtain definite particle sizes for the fine material. This device gives the fraction of particles with an apparent spherical diameter. The Coulter was found to be the most versatile method overall in a review by Swift, et al **(164)**; however, this method does not provide settling information. Another method for determining the particle size distribution of a very fine fraction is through the use of electron microscopy **(165)**. The collection technique for the very fine materials can result in aggregation to larger colloidal structures **(165-168)**. Comparisons of particle sizing methods have shown that some produce similar results and others do not. These differences might be attributed to differences in the particle property being measured. That is, the Malvern Laser Sizer and Electrozone Particle Counter are sizing techniques, and the hydrophotometer and SediGraph determine sedimentation diameter based on particle settling **(154, 169-171)**. It is preferable to use a method that incorporates particle settling as a measure, as opposed to strictly sediment sizing.

12.6 Various methods have been recommended for determining the bioavailable fractions of metals in sediments **(71, 172-174)**. One extraction procedure, cation-exchange capacity, provides information relevant to metal bioavailability studies **(140)**. Amorphic oxides of iron and manganese, and reactive particulate carbon, have been implicated as the primary influences on the metal sorption potential in sediments **(75, 173, 175-177)**. The measurement of acid volatile sulfide (AVS) and divalent metal concentrations associated with AVS extraction provides insight into metals availability in anaerobic sediments **(71)**. Easily extractable fractions are usually removed with cation displacing solutions, for example, neutral ammonium acetate, chloride, sodium acetate, or nitrate salts **(178)**. However, the extraction of saltwater or calcareous sediments, is often complicated by complexation effects or the dissolution of other sediment components **(174, 179)**. Other extractants and associated advantages and disadvantages have been discussed **(174, 177, 180, 181)**. Some extractants that have been used successfully in evaluations of trace metals in nondetrital fractions of sediments are EDTA or HCl **(174, 182, 183)**. Metal partitioning in sediments might be determined by using sequential extraction procedures that fractionate the sediments into several components such as interstitial water, ion exchangeable, easily reducible organic, and residual sediment components **(95, 180, 184, 185)**. Unfortunately, no one method is clearly superior to the others at this time **(179)**. This might partly be due to site-specific characteristics that influence bioavailability, for example, desorption and equilibration processes.

12.7 pH is important for many chemicals and can be measured directly **(13)** or in a 1 to 1 mixture of sediment and soil to water **(186)**.

12.8 Eh measures are particularly important for metal speciation and for determining the extent of sediment oxidation. Redox gradients in sediments often change rapidly over a small depth and are disturbed easily. Care must be taken in probe insertion to allow equilibration to occur when measuring Eh. These measurements are potentiometric and measured with a platinum electrode relative to a standard hydrogen electrode **(13)**.

12.9 Biochemical oxygen demand and chemical oxygen demand might provide useful information in some cases **(13)**. Sediment oxygen demand might also be a useful descriptor; however, a wide variety of methods exists **(86, 137-190)**.

12.10 The analysis of toxicants in sediments is generally performed by standard methods such as those of the Environmental Protection Agency (EPA) **(4, 13, 191)**. Acid digests are necessary for bound metal extraction. Soxhlet extraction is generally best for organics but depends on the extraction parameters **(192, 193)**. Concentrations are generally reported on a dry weight or organic carbon basis. The sample size requirements for chemical and physical analysis are generally as follows: organics, 250 g (wet); metals, 100 g; ammonia, 100 g; grain size, 500 g; total organic carbon, 50 g; and total solids, 50 g.

13. Manipulations

13.1 Manipulation of sediments is often required to yield consistent material for toxicity testing and laboratory experiments. The manipulations reviewed in this section are as follows: (*1*) mixing, (*2*) spiking, (*3*) sieving, (*4*) dilutions for concentration-effect determinations, (*5*) elutriates, and (*6*) capping. See 9.7 for discussions of subsampling, compositing, or homogenization.

13.2 Mixing of sediments is conducted to produce an homogeneous sample that is uniform in color, texture, and moisture and that yields precise results in replicate determination of toxicity. For field-collected sediments, the sediment quality will be influenced by the depth of sampling, depth of biological activity, contaminant solubility and partitioning characteristics, and depth of the contaminant concentration peak, which is dependent on the historical contamination and sedimentation rates for the study site. As a result, mixing of various layers of sediments might result in either the dilution or enhancement of concentrations (see Section 10 for additional relevant discussions). Hand mixing can be accomplished by blending with a spatula **(45, 61, 194-198)**, rolling the sediment out flat on a sheet of plastic or pre-combusted foil and tumbling by raising each corner of the sheet in succession, or by coning (mounding the sediment) followed by quartering and remixing **(199, 200)**. A variety of mechanical mixers, such as a hand-held drill equipped with a polypropylene stirrer (for example, **62, 201**), a rolling mill **(201-203)**, or gyro-rotary and Eberbach shakers **(62)**, have also been used. The mixing time for sediments that differ in color, texture, moisture, volume, and layering will vary but will generally be in the range from one to several minutes **(201, 204)**. Mechanical mixing may alter the particle size distribution. It is therefore recommended that the particle size be determined prior to and following the mixing process in order to monitor potential changes in grain size due to the mixing process. Regardless of the mixing method, the efficiency of mixing must also be demonstrated by determining the coefficients of variation **(205)** for chemical or physical analyses from replicated samples (see 13.5 for further discussion).

13.3 *Spiking*—Whole sediments may be spiked with specific chemicals in order to determine the effects of single toxicants or mixtures of toxicants on biota **(45, 74, 76, 142, 200, 206-210)**. The primary methods used to spike sediments with contaminants involve dry- and wet-spiking techniques. Air-dried sediments have been spiked successfully with organic compounds in dose-response toxicity tests **(45, 143, 208, 210, 211)**. However, air drying may result in losses of volatile compounds as well as changes in sediment characteristics, especially particle size. The presence of air and air drying have also been shown to change metal availability and complexation, and dry-spiking is therefore not recommended. Wet-spiking techniques are currently the most acceptable for the preparation of a spiked sediment, and several techniques have been used, depending on the chemical used in spiking **(74, 200, 201, 206, 207, 210, 212, 213)**.

13.4 Wet-spiking methodologies differ mainly in the amount of water present in the mixture during spiking, solvent used to apply the toxicant, and method of mixing. In many cases, the compound is either coated on the walls of the flask, and an aqueous slurry (sediment and water in various proportions) added, or the carrier-containing mixture is added directly to the slurry. When the sediment-to-water ratio is adjusted for optimal mixing, sediments that are too dense to mix by slurrying in water have been mixed successfully using the rolling mill **(201-203)**. In addition to the rolling mill technique, thorough mixing of spiked sediments has been accomplished using Eberback and gyro-rotary shakers **(62)**. A chemical can also be added to the water overlying the sediment and allowed to sorb with no mixing **(65, 214-220)**. A carrier has occasionally been added directly to sediment **(41, 76-78, 169, 211, 213, 221-224)** and the carrier evaporated before the addition of water, leaving the chemical in a crystalline form. This approach does not seem to result in compounds being sorbed to sediment at the same sites as dosing under aqueous conditions **(205)**. Care should be taken to ensure complete and homogenous mixing (see 13.2) no matter what technique is used for spiking. In addition, chemical analyses should be conducted to ensure that spiking is uniform in the mixed material (see 13.5). The mixing time following spiking should be limited to a few minutes or hours **(1-26)**, and temperatures should be kept to a minimum (for example, 4°C) due to the rapid alterations that may occur in the sediment's physicochemical and microbiological characteristics that could alter bioavailability and toxicity. The mixing time might be extended for recalcitrant organics and some metals (for example, cadmium and copper) without adverse effects (see Sections 9-12 for additional discussion).

13.5 One of the most important criteria for the choice of both the mixing methodology and chemical used in the preparation of a spiked sediment is that homogeneous mixing occurs within the substrate. Ditsworth, et al **(201)** found that coefficients of variation (CVs) ranged from 2.2 to 10.9 % (mean of 4.8 %) for cadmium levels in cadmium-spiked sediment samples collected along a longitudinal axis of an horizontally lying mixing jar **(201)**. The CVs did not increase with nominal cadmium levels (as $CdCl_2$, range from 3.5 to 14 mg/kg) added to the sediment. Significant differences in cadmium concentration existed among sampling locations within jars in some cases. Regarding organics, Ditsworth, et al **(201)** reported that mixing fluoranthene into one jar of sediment using the rolling-jar technique provided a CV of 11.5 %

between sample locations within the jar, and no significant effect of the sample location was found. Good mixing efficiency for fluoranthene was also shown by Suedel, et al **(213)** with a CV of 10.3 % when the chemical was added directly to sediment, the carrier evaporated, and the sediment mixed by hand for 60 s before the addition of test water. Landrum and coworkers have found the following CVs for sediments at various concentrations in different experiments and using the slurry technique: pyrene, 4.8 to 6.9 %; phenanthrene, 4.7 to 9.3 %; BaP, 5.8 ± 3.2 %; hexachlorobiphenyl, 7.8 ± 4.5 %; and tetrachlorobiphenyl, 9.1 ± 5.0 % (**200, 210**, unpublished data). CVs should be ≤20 % for the homogeneity of mixing to be considered valid **(225)**. However, it should be noted that the concentrations of total chemical determined in the sediment matrix do not reflect the bioavailable fraction of the chemical.

13.6 The spiking method to be used is contingent on the study objectives, sediment type, and compound(s) of interest. For example, when attempting to mimic in situ conditions, sediment cores should be spiked by adding an aqueous or suspended sediment solution of toxicants to the overlying water column, as would occur in the natural environment; or, when investigating the dredging effects or conditions of sediment perturbation where toxicant sorption processes are accelerated, mixing toxicants into sediment slurries may be advantageous. When investigating the source of sediment toxicity or interactive effects of sediment toxicants, it is useful to spike both the reference and control sediments with the toxicant of concern present in the test sediment.

13.7 Organic compounds are generally added by means of a carrier solvent, such as acetone or methanol, to ensure that they are soluble and remain in solution during mixing. Word, et al **(114)** compared several sediment-labeling techniques using methylene chloride, ethanol, and glycine as carriers. They found that glycine was superior when mixed with sediment for seven days. The use of a polar water-soluble carrier such as methanol has little effect on the partitioning of nonpolar compounds to dissolved organic matter at concentrations up to 15 % carrier by volume **(226)**. However, another study shows that changes in partitioning by a factor of approximately two might well occur with 10 % methanol as a cosolvent for anthracene sorption **(227)**. Caution should thus be taken to minimize the amount of carrier used. Metals are added in aqueous solutions while organic compounds are generally added in an organic carrier.

13.8 A variety of methods have been used to spike sediments with metals, but the two principal categories of methods are as follows: metal addition directly to the sediment, which is mixed and then water added **(62, 87, 207, 212, 228)**; and addition of the metal to the overlying waters **(74, 229-231)**.

13.9 Highly volatile compounds have been spiked into sediments using cosolvents followed by shaking in an aqueous slurry. Immediate testing in covered flow-through systems is recommended **(232)** when highly volatile compounds are used.

13.10 If a solvent other than water is used, both a sediment solvent control and sediment negative control or reference sediment, or both, must be included in the test. The solvent control must contain the highest concentration of solvent present and must use solvent from the same batch used to make the stock solution.

13.11 Once a sediment has been spiked with the toxicant of choice, it is necessary to allow the mixture to reach equilibrium before commencing a whole-sediment toxicity test. Equilibrium is defined as in equilibrium partitioning and refers to the assumption that an equilibrium exists between the chemical sorbed to the particulate sediment components and the pore water **(233)**. The equilibration times and storage procedures for spiked sediments vary widely among studies **(234)**, and there has been no attempt to standardize them. This is partly because accurate methods for measuring true equilibrium scientifically (that is, accurately isolating interstitial water and measuring the freely dissolved fraction of the compound of interest) are currently lacking, and little information exists on how long it will take for equilibrium to be established for any compound. In addition, the time to reach equilibrium will differ for compounds and sediments of differing characteristics. For metals, the time could be as short as 24 h **(230, 235)** or as long as 120 days. Similarly, for organics, the time allowed for the sediment and water to equilibrate has been as short as 24 h **(236)** or as long as 5 weeks **(203)**. The duration of contact between the toxicant and sediment particles can affect both the partitioning and bioavailability of the toxicant. For example, Landrum, et al **(200)** found that the partitioning of pyrene and phenanthrene between sediment particles and interstitial water increased significantly, whereas the uptake rate coefficients for the amphipod, *Diporeia sp*., decreased significantly for both chemicals as the contact time increased. This effect occurs apparently because of an initial rapid labile sorption followed by movement of the toxicant into resistant sorption sites or in the particle **(237-239)**. The contact time can be important when spiking sediments because of the kinetically controlled changes in the partitioning that results in changes in bioavailability **(200, 210, 227, 240)**. Bounds on the sorption time can be estimated from the partition coefficient for the sediment following the calculations in Karickhoff and Morris **(238)**. In addition, it is important to recognize that the quantity of toxicant spiked might exceed the complexation capacity of the test sediment system and not allow reactions to attain equilibrium. These phenomena will complicate the interpretation of test results **(62, 180)**. Until more definitive information is generated, it is recommended that a standard equilibration time (for example, 2 weeks at 4°C) be established between the initial contact of the contaminant with the sediment and the initiation of toxicity tests.

13.12 The organic carbon content of sediments may be one of the most important characteristics affecting the biological availability of contaminants. Modifications of the carbon content have therefore been made in many studies. Methods for modification include dilution with clean sand **(44, 45, 51, 232)** or humics **(224)**, and other organics such as sheep manure **(41, 64)**, or the addition of organic detritus such as feces of *Crassostrea gigas* or *Callianassa californiensis* **(203)**. Such dilutions also change the particle composition and size distribution of the particles; results from such experiments should thus be interpreted with care. The organic carbon content has also been altered by the use of combustion **(41, 241)**. Combustion may alter the type of carbon as well as oxidize some of the inorganic components, thus altering the characteristics of the sediment greatly.

13.13 Although the sieving of field-collected sediments is known to disrupt chemical equilibrium, such manipulations may be necessary before toxicity tests are performed **(18, 41, 47-49, 61, 64, 143, 144, 206, 208, 209, 211, 217, 223, 242)**. Justifications for sieving include the removal of large stones and other debris; removal of endemic species; improved sample homogeneity and replication; improved counting efficiency of organisms; increased ease of sediment handling and subsampling; and ability to study the influence of particle size on toxicity, bioavailability, or contaminant partitioning. Sediments can be either wet sieved **(62, 194, 198, 199, 243)** or pressure-sieved **(244)**. Wet sieving involves agitating or swirling the sieve containing sediment in water so that particles smaller than the selected mesh size are washed through the sieve into a container. The sieve may be placed on a mechanical shaker, or the sediments on the screen can be stirred with a nylon brush **(199)**, to facilitate the process. Alternatively, the particles may be washed through the sieve with a small volume of running water **(245)**. Particles retained in the sieve (the coarse fraction) are examined and retained if they are of interest to the study. Pressure sieving involves the pressing of sediment particles through a sieve having an appropriate mesh size with a mechanical, piston-type arrangement, or with a flat-surfaced, hand-held tool. This technique works well with sediments containing few stones or other large objects and with a low to moderate clay content. Also, the method is applied best using sieves with mesh sizes >0.50 mm. Sieves used in toxicity tests can be constructed of stainless steel or plastic (for example, polyethylene, polypropylene, nylon, and PTFE), with mesh sizes varying from 0.25 to 2.0 mm **(62, 114, 195, 198, 208, 209, 244, 246)**. The mesh size used most frequently for sieving is 1.0 mm, but the choice of mesh size is dependent on the objectives of the study and whether indigenous organisms must be removed from the test sediment (see 13.14 and 13.15).

13.14 The sieving of sediments may also (increase or decrease) the concentrations of contaminants contained in test sediments. Particles and their attendant contaminant loads may be either concentrated or removed. Also, sieving may disrupt chemical equilibria through the volatilization or modification of sorption and desorption characteristics. For example, Day, et al **(247)** found that sieving contaminated sediment through 250-µm mesh decreased concentrations of PCBs and PAHs as much as four-fold. Surface areas (in relation to the weight of the sample) and sorptive capacities are higher in fine-grained sediments (that is, clay and silt), and organic carbon concentrations as well as toxic chemicals thus tend to be higher in these sediments. Measuring size fractions of less than 63 µm has been recommended in contaminant studies with sediments, particularly for metals **(225, 248)**. In studies of metal concentrations in sediments, normalizing to the< 63-µm size fractions was superior for describing metal binding in sediments, compared to sediment concentrations normalized to dry weight, by organic carbon content, or corrected by a centrifugation procedure **(225)**. Small-size fractions are characteristic of depositional areas in aquatic systems; however, the sieving of sediments from non-depositional sites to obtain the fine fraction might alter the sediment characteristics significantly. It is recommended that sediments not be sieved, unless the sediments contain excessive quantities of large organic debris or if the contamination is being normalized to a specific grain size.

13.15 The suspected presence of endemic organisms that will interfere with the results of chronic toxicity tests (for example, oligochaete worms, leeches, chironomids, etc.) will also necessitate the sieving of some field-collected sediments.

13.16 The presence of endemic species in sediments used in toxicity tests has been shown to complete the interpretation of acute and chronic endpoints **(196, 247, 249, 250)**. Swartz, et al **(202)** demonstrated that the optimum mesh size for the removal of endemics was 0.50 mm for marine sediments. In freshwater sediments, the removal of large predators such as leeches can be accomplished by hand-picking with tweezers, but species of invertebrates that are morphologically similar to or in competition for space and food with species used in toxicity tests, or both, can be eliminated only by sieving with a mesh size of ≤0.25 mm **(246, 248)**. In order to eliminate potential interferences from endemic species in freshwater samples, but limit the unnecessary sieving of sediments, it is recommended that a subsample of field-collected sediment be examined under low magnification using a stereomicroscope and, if coccoons, juvenile instars, or adults of endemic species such as oligochaete worms or chironomids are noted, that sieving of test sediments be conducted.

13.17 Methods other than sieving to inhibit endemic biological activity in field-collected sediments include autoclaving, freezing, and gamma irradiation of sediments **(247, 251)**. Caution is required in the use of these techniques, depending on the objectives of the study and test species to be used in the subsequent toxicity test. For example, Day, et al **(247)** found that survival of the amphipod, *Hyalella azteca*, was reduced significantly in any sediment that was frozen, autoclaved, or gamma irradiated. The reasons for this response are unknown but may relate to changes in the physical structure of sediments during these manipulations, an increased bioavailability of toxic compounds within the sediment matrice due to changes in chemical equilibria, or a reductions in sources of food for *H. azteca* due to sterilization. Malueg, et al **(61)** found that freezing sediment attenuated the release of total and soluble copper from the sediment into the overlying water. In contrast to the studies with *H. azteca*, growth of the chironomid, *Chironomus riparius*, and reproduction of the tubificid worm, *Tubifex tubifex*, were enhanced in sediments that had been sterilized by autoclaving or gamma irradiation **(247)**. Tubificid worms and chrionomids feed on organic material as well as particles of sediment within the benthos, and the sterilization of sediments may increase organic material **(252)**, thereby providing more food for the test organisms and thus better growth. Other sterilization techniques have included the use of antibiotics such as streptomycin and ampicillin (Danso, et al, 1973; Burton, et al, 1987) or the addition of chemical inhibitors such as $HgCl_2$ or sodium azide. Information on the effects of sediments that have received these treatments to toxicity test responses is not available. Some antibiotics are labile and light sensitive or bind readily to organic matter, so their use in all situations may not be appropriate. Mercuric chloride appears to be superior to sodium azide as a bacteriocide. It is crucial that a sterility control be incorporated in studies requiring sterility.

13.18 Diluting a test sediment with a clean, non-contaminated sediment has been suggested as an approach in order to obtain concentration-effects information in solid phase toxicity testing **(234, 253, 254)**. Such dilutions have been performed with reference sediments **(236, 244, 255-258)** or clean sand **(258, 259)**. Dilutions with test sediment have generally led to reductions in the toxicity of the diluted material relative to the test sediment. However, the toxicity decreased and then increased subsequently for some sediments **(258)** when sand was used as a diluent, although the sand alone was not toxic compared to controls. The mechanism for this effect is not known. The dilutions were generally mixed to visual homogeneity where described, and the only report of a definitive storage time after mixing was for 10 days **(244, 259)** and the temperature for storing diluted sediments was 4°C **(244)**. No definitive testing has been performed on the appropriate length of storing dilutions. The actual amount of dilution can be estimated by determining the fraction of fine material and organic carbon content in the reference sediment, test sediment, and diluted material **(258)**. Little information remains on the most appropriate method for diluting test sediments to obtain graded contaminant concentrations. Little is known concerning the role of sediment composition, equilibrium time, and alteration of chemistry during mixing on the exposure to the test sediment contaminants in the diluted material. A clean, noncontaminated sediment should be used as the diluent. This sediment should optimally have characteristics similar to the test sediment, such as organic matter and carbon concentration and particle size distribution, and should not contain elevated levels of the toxicants. Pure sand does not appear to be an appropriate dilution material because of the changes in toxicity with differing dilutions.

13.19 Elutriate tests, or aqueous extractions of resuspended sediments, have been conducted routinely **(92, 260, 261)**. The method of elutration was originally developed **(262)** to simulate processes that might disturb the sediment and thus bring contaminants into the water column, that is, dredging activities, but the method has been adapted further to evaluate the effects of other common events that disrupt sediments and affect water quality, such as bioturbation and storms **(24, 90)**. Elutriates are generally prepared by combining various mixtures of water and sediment (usually 4:1 ratio, v/v) and shaking, bubbling, or stirring the mixture for 1 h **(90, 92, 261, 263)**. The water phase is then separated from the sediment by centrifugation, and the supernatant is used in various toxicity tests (for example, fathead minnow, *Pimephales promelas*; bioluminescence assay, *Photobacterium phosphoreum*; and sea urchin (*Arbacia punctulata*), fertilization test). Filtration of the supernatant through filters (0.45 to 1.2 µm) may be necessary when the elutriate is used in some toxicity tests such as the algal growth assay with *Selenastrum capricornutum*. However, as discussed in previous sections, filtration can remove toxicity due to the sorption of dissolved chemicals to the filtration membrane and retention of colloids. Elutriates have generally been found to be less toxic than bulk sediments or interstitial water fractions **(90, 92)** to various biota, but there have been isolated cases in which resuspension increased the bioavailability of toxicants in the water column. Partitioning to organic colloids in the interstitial water has been suggested as a possible explanation for the discrepancies between suspended-phase and interstitial water exposures **(90)**. Toxicity may be affected significantly by the method of elutriation; data comparisons should therefore be made only where standardized elutriate methods were used.

13.20 The remediation of sediment might include capping the contaminated sediments with clean sediments. The laboratory design of such experiments should vary the depth of both the contaminated sediments and the capping sediment layers to evaluate contaminant transport by means of physiochemical and biological (bioturbation) processes.

14. Quality Assurance

14.1 The QA guidelines **(9, 10, 39, 57, 264)** should be followed. The QA considerations for sediment modeling, QA-QC plans, statistical analyses (for example, sample number and location), and sample handling have been addressed in depth **(57)**.

14.2 Sediment heterogeneity significantly influences studies of sediment quality, contaminant distribution, and both benthic invertebrate and microbial community effects. Spatial heterogeneity might result from numerous biological, chemical, and physical factors and should be considered both horizontally (such as on the sediment surface) and vertically (that is, depth). Accumulation areas with similar particle size distributions might yield significantly different toxicity patterns when subsampled **(73, 265)**; an adequate number of replicates should therefore be processed to determine site variance. When determining site variance, one should consider within sample (that is, subsample) variance, analytical variance (for example, chemical or toxicological), and the sampling instruments' accuracy and precision. A sampling design can be constructed after these considerations that addresses the resource limitations and study objectives.

14.3 As stated in previous sections, the methodological approach used, such as number of samples, will depend on the study objectives and sample characteristics. There are a number of references available for information on sediment heterogeneity; splitting; compositing; controls; or determining sample numbers, sampler accuracy and precision, and resource requirements **(10, 11, 57, 80, 225, 266, 267)**.

14.4 Quality assurance is an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that an environmental assessment is of the type and quality necessary. Quality control is the overall system of technical activities that measures and controls the quality of the assessment. The primary mechanism for ensuring that there is an adequate QA-QC program is through a Quality Assurance Project Plan (QAPP). This formal document describes, in detail, the necessary QA and QC procedures that are implemented to ensure that the results of the assessment will satisfy the stated performance criteria. This process is described in detail in

USEPA **(5)**. The QAPP describes the following: project description; project organization and responsibilities; QA objectives for the measurement data (including data quality objectives, precision, accuracy, test acceptability, representativeness, completeness, and comparability; sampling; analytical or test procedures (standard operating procedures); sample custody procedures; calibration procedures and frequency; internal QC checks and frequency; performance and system audits; analytical procedures; data reduction, validation, assessment, and reporting procedures; preventive maintenance procedures and schedules; corrective action; and QA reports to management. Refer to the appropriate standard test method guidance (for example, ASTM, USEPA, and APHA) for acceptable quality control limits for test measurements (for example, toxicity assay performance criteria, analytical precision, accuracy, completeness, and method detection limit).

15. Report

15.1 *Documentation*—Include the following information, either directly or by reference to existing documents, in the record of sediment collection, storage, handling, and manipulation. Published reports should contain enough information to identify the methodology used and quality of the results clearly. Specific information should include the following:

15.1.1 Name of the test and investigator(s); name and location of the sample station and test laboratory; field conditions (for example, water depth, sampler penetration depth in sediment, sediment characteristics, collection and storage methods, and dates of starting and ending of sampling and sediment manipulation;

15.1.2 Source of the control, reference, or test sediment; method for handling, storage, and disposal of the sediment;

15.1.3 Source of the water; its chemical characteristics; a description of any pretreatment;

15.1.4 Methods used for, and results (with confidence limits) of, physical and chemical analyses of the sediment; and

15.1.5 Anything unusual concerning the study, any deviation from these procedures, manipulations, and any other relevant information.

16. Keywords

16.1 characterization; collection; manipulation; sediment; storage; toxicity; transport

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