Designation: E1391 – 03 (Reapproved 2014)

Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates[1]

This standard is issued under the fixed designation E1391; 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 marine, estuarine, and freshwater sediments, for use in laboratory sediment toxicity evaluations and describes samplers that can be used to collect sediment and benthic invertebrates (Annex A1). This standard is not meant to provide detailed 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 methods are reviewed in this guide. A statement on the consensus approach then follows this review of the methods. This consensus approach has been included in order to foster consistency among studies. It is anticipated that recommended methods and this guide will be updated routinely to reflect progress in our understanding of sediments and how to best study them. This version of the standard is based primarily on a document developed by USEPA (2001 (**1**))[2] and by Environment Canada (1994 (**2**)) as well as an earlier version of this standard.

1.2 Protecting sediment quality is an important part of restoring and maintaining the biological integrity of our natural resources as well as protecting aquatic life, wildlife, and human health. Sediment is an integral component of aquatic ecosystems, providing habitat, feeding, spawning, and rearing areas for many aquatic organisms (MacDonald and Ingersoll 2002 a, b (**3**)(**4**)). Sediment also serves as a reservoir for contaminants in sediment and therefore a potential source of contaminants to the water column, organisms, and ultimately human consumers of those organisms. These contaminants can arise from a number of sources, including municipal and industrial discharges, urban and agricultural runoff, atmospheric deposition, and port operations.

1.3 Contaminated sediment can cause lethal and sublethal effects in benthic (sediment-dwelling) and other sediment-associated organisms. In addition, natural and human disturbances can release contaminants to the overlying water, where pelagic (water column) organisms can be exposed. Sediment-associated contaminants can reduce or eliminate species of recreational, commercial, or ecological importance, either through direct effects or by affecting the food supply that sustainable populations require. Furthermore, some contaminants in sediment can bioaccumulate through the food chain and pose health risks to wildlife and human consumers even when sediment-dwelling organisms are not themselves impacted (Test Method E1706).

1.4 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. Ingersoll et al. (1997 (**5**)), and Wenning and Ingersoll (2002 (**6**)).

1.5 This guide is arranged as follows:

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[1] This guide is under the jurisdiction of ASTM Committee E50 on Environmental Assessment, Risk Management and Corrective Action and is the direct responsibility of Subcommittee E50.47 on Biological Effects and Environmental Fate.

Current edition approved Oct. 1, 2014. Published May 2015. Originally approved in 1990. Last previous edition approved in 2008 as E1391 – 03(2008). DOI: 10.1520/E1391-03R14.

[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

*A Summary of Changes section appears at the end of this standard

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1.6 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.

1.7 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.

1.8 *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 requirements prior to use.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1067 Test Methods for Acidity or Alkalinity of Water
D1126 Test Method for Hardness in Water
D1129 Terminology Relating to Water
D1426 Test Methods for Ammonia Nitrogen In Water
D3976 Practice for Preparation of Sediment Samples for Chemical Analysis
D4387 Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates (Withdrawn 2003)[4]
D4822 Guide for Selection of Methods of Particle Size Analysis of Fluvial Sediments (Manual Methods)
D4823 Guide for Core Sampling Submerged, Unconsolidated Sediments
E729 Guide for Conducting Acute Toxicity Tests on Test Materials with Fishes, Macroinvertebrates, and Amphibians
E943 Terminology Relating to Biological Effects and Environmental Fate
E1241 Guide for Conducting Early Life-Stage Toxicity Tests with Fishes
E1367 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates
E1525 Guide for Designing Biological Tests with Sediments
E1611 Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids
E1688 Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates
E1706 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates
IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

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 E729 and Test Method E1706, Terminologies D1129 and E943, and Classification D4387; for an explanation of units and symbols, refer to IEEE/ASTM SI 10.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *site, n*—a study area comprised of multiple sampling station.

3.2.2 *station, n*—a location within a site where physical, chemical, or biological sampling or testing is performed.

4. Summary of Guide

4.1 This guide provides a review of widely used methods for collecting, storing, characterizing, and manipulating sediments for toxicity or bioaccumulation testing and also describes samplers that can be used to collect benthic invertebrates. Where the science permits, recommendations are provided on which procedures are appropriate, while identifying their limitations. This guide addresses the following general topics: (*1*) Sediment monitoring and assessment plans (including developing a study plan and a sampling plan), (*2*) Collection of whole sediment samples (including a description of various sampling equipment), (*3*) Processing, transport and storage of sediments, (*4*) Sample manipulations (including sieving, formulated sediments, spiking, sediment dilutions, and preparation of elutriate samples), (*5*) Collection of interstitial water (including sampling sediments in situ and ex situ), (*6*) Physico-chemical characterizations of sediment samples, (*7*) Quality assurance, and (*8*) Samplers that can be used to collect sediment or benthic invertebrates.

5. Significance and Use

5.1 Sediment toxicity evaluations are a critical component of environmental quality and ecosystem impact assessments, and are used to meet a variety of research and regulatory

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

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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.

5.2 Sediment quality assessment is an important component of water quality protection. Sediment assessments commonly include physicochemical characterization, toxicity tests or bioaccumulation tests, as well as benthic community analyses. The use of consistent sediment collection, manipulation, and storage methods will help provide high quality samples with which accurate data can be obtained for the national inventory and for other programs to prevent, remediate, and manage contaminated sediment.

5.3 It is now widely known that the methods used in sample collection, transport, handling, storage, and manipulation of sediments and interstitial waters can influence the physicochemical properties and the results of chemical, toxicity, and bioaccumulation analyses. Addressing these variables in an appropriate and systematic manner will provide more accurate sediment quality data and facilitate comparisons among sediment studies.

5.4 This standard provides current information and recommendations for collecting and handling sediments for physicochemical characterization and biological testing, using procedures that are most likely to maintain in situ conditions, most accurately represent the sediment in question, or satisfy particular needs, to help generate consistent, high quality data collection.

5.5 This standard is intended to provide technical support to those who design or perform sediment quality studies under a variety of regulatory and non-regulatory programs. Information is provided concerning general sampling design considerations, field and laboratory facilities needed, safety, sampling equipment, sample storage and transport procedures, and sample manipulation issues common to chemical or toxicological analyses. Information contained in this standard reflects the knowledge and experience of several internationally-known sources including the Puget Sound Estuary Program (PSEP), Washington State Department of Ecology (WDE), United States Environmental Protection Agency (USEPA), US Army Corps of Engineers (USACE), National Oceanic and Atmospheric Administration (NOAA), and Environment Canada. This standard attempts to present a coherent set of recommendations on field sampling techniques and sediment or interstitial water sample processing based on the above sources, as well as extensive information in the peer-reviewed literature.

5.6 As the scope of this standard is broad, it is impossible to adequately present detailed information on every aspect of sediment sampling and processing for all situations. Nor is such detailed guidance warranted because much of this information (for example, how to operate a particular sampling device or how to use a Geographical Positioning System (GPS) device) already exists in other published materials referenced in this standard.

5.7 Given the above constraints, this standard: (*1*) presents a discussion of activities involved in sediment sampling and sample processing; (*2*) alerts the user to important issues that should be considered within each activity; and (*3*) gives recommendations on how to best address the issues raised such that appropriate samples are collected and analyzed. An attempt is made to alert the user to different considerations pertaining to sampling and sample processing depending on the objectives of the study (for example, remediation, dredged material evaluations or status and trends monitoring).

5.8 The organization of this standard reflects the desire to give field personnel and managers a useful tool for choosing appropriate sampling locations, characterize those locations, collect and store samples, and manipulate those samples for analyses. Each section of this standard is written so that the reader can obtain information on only one activity or set of activities (for example, subsampling or sample processing), if desired, without necessarily reading the entire standard. Many sections are cross-referenced so that the reader is alerted to relevant issues that might be covered elsewhere in the standard. This is particularly important for certain chemical or toxicological applications in which appropriate sample processing or laboratory procedures are associated with specific field sampling procedures.

5.9 The methods contained in this standard are widely applicable to any entity wishing to collect consistent, high quality sediment data. This standard does not provide guidance on how to implement any specific regulatory requirement, or design a particular sediment quality assessment, but rather it is a compilation of technical methods on how to best collect environmental samples that most appropriately address common sampling objectives.

5.10 The information presented in this standard should not be viewed as the final statement on all the recommended procedures. Many of the topics addressed in this standard (for example, sediment holding time, formulated sediment composition, interstitial water collection and processing) are the subject of ongoing research. As data from sediment monitoring and research becomes available in the future, this standard will be updated as necessary.

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

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complicates interpretations of treatment effects, causative factors, and in situ comparisons. Individual sections address specific interferences.

7. Apparatus

7.1 A variety of sampling, characterization, and manipulation methods exist using different equipment. These are reviewed in Sections 10 – 14.

7.2 *Cleaning*—Equipment used to collect and store sediment samples, equipment used to collect benthic invertebrate samples, equipment used to prepare and store water and stock solutions, and equipment used to expose test organisms should be cleaned before use. All non-disposable sample containers, test chambers, and other equipment that have come in contact with sediment should be washed after use in the manner described as follows to remove surface contaminants (Test Method E1706). See 10.4 for additional detail.

8. Safety Hazards

8.1 *General Precautions:*

8.1.1 Development and maintenance of an effective health and safety program in the laboratory requires an ongoing commitment by laboratory management and includes: (*1*) the appointment of a laboratory health and safety officer with the responsibility and authority to develop and maintain a safety program, (*2*) the preparation of a formal, written health and safety plan, which is provided to each laboratory staff member, (*3*) an ongoing training program on laboratory safety, and (*4*) regular safety inspections.

8.1.2 Collection and use of sediments may involve substantial risks to personal safety and health. Chemicals in field-collected sediment may include carcinogens, mutagens, and other potentially toxic compounds. Inasmuch as sediment testing is often started before chemical analyses can be completed, worker contact with sediment needs to be minimized by: (*1*) using gloves, laboratory coats, safety glasses, face shields, and respirators as appropriate, (*2*) manipulating sediments under a ventilated hood or in an enclosed glove box, and (*3*) enclosing and ventilating the exposure system. Personnel collecting sediment samples and conducting tests should take all safety precautions necessary for the prevention of bodily injury and illness that might result from ingestion or invasion of infectious agents, inhalation or absorption of corrosive or toxic substances through skin contact, and asphyxiation because of lack of oxygen or presence of noxious gases.

8.1.3 Before beginning sample collection and laboratory work, personnel should determine that all required safety equipment and materials have been obtained and are in good condition.

8.2 *Safety Equipment:*

8.2.1 *Personal Safety Gear*—Personnel should use safety equipment, such as rubber aprons, laboratory coats, respirators, gloves, safety glasses, face shields, hard hats, safety shoes, water-proof clothing, personal floatation devices, and safety harnesses.

8.2.2 *Laboratory Safety Equipment*—Each laboratory should be provided with safety equipment such as first-aid kits, fire extinguishers, fire blankets, emergency showers, and eye wash stations. Mobile laboratories should be equipped with a telephone to enable personnel to summon help in case of emergency.

8.3 *General Laboratory and Field Operations:*

8.3.1 Special handling and precautionary guidance in Material Safety Data Sheets (MSDS) should be followed for reagents and other chemicals purchased from supply houses.

8.3.2 Work with some sediments may require compliance with rules pertaining to the handling of hazardous materials. Personnel collecting samples and performing tests should not work alone.

8.3.3 It is advisable to wash exposed parts of the body with bactericidal soap and water immediately after collecting or manipulating sediment samples.

8.3.4 Strong acids and volatile organic solvents should be used in a fume hood or under an exhaust canopy over the work area.

8.3.5 An acidic solution should not be mixed with a hypochlorite solution because hazardous fumes might be produced.

8.3.6 To prepare dilute acid solutions, concentrated acid should be added to water, not vice versa. Opening a bottle of concentrated acid and adding concentrated acid to water should be performed only under a fume hood.

8.3.7 Use of ground-fault systems and leak detectors is strongly recommended to help prevent electrical shocks. Electrical equipment or extension cords not bearing the approval of Underwriter Laboratories should not be used. Ground-fault interrupters should be installed in all "wet" laboratories where electrical equipment is used.

8.3.8 All containers should be adequately labeled to indicate their contents.

8.3.9 A clean and well-organized work place contributes to safety and reliable results.

8.4 *Disease Prevention*—Personnel handling samples which are known or suspected to contain human wastes should be immunized against hepatitis B, tetanus, typhoid fever, and polio. Thorough washing of exposed skin with bacterial soap should follow handling of samples collected from the field.

8.5 *Safety Manuals*—For further guidance on safe practices when handling sediment samples and conducting toxicity tests, check with the permittee and consult general industrial safety manuals including(**7**),(**8**).

8.6 *Pollution Prevention, Waste Management, and Sample Disposal*—Guidelines for the handling and disposal of hazardous materials should be strictly followed (Guide D4447). The Federal Government has published regulations for the management of hazardous waste and has given the States the option of either adopting those regulations or developing their own. If States develop their own regulations, they are required to be at least as stringent as the Federal regulations. As a handler of hazardous materials, it is your responsibility to know and comply with the pertinent regulations applicable in the State in which you are operating. Refer to the Bureau of National Affairs Inc. (**9**) for the citations of the Federal regulations.

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9. Sediment Monitoring and Assessment Study Plans

9.1 Every study site (for example, a study area comprised of multiple sampling stations) location and project is unique; therefore, sediment monitoring and assessment study plans should be carefully prepared to best meet the project objectives (MacDonald et al. 1991(**10**); Fig. 1).

9.2 Before collecting any environmental data, it is important to determine the type, quantity, and quality of data needed to

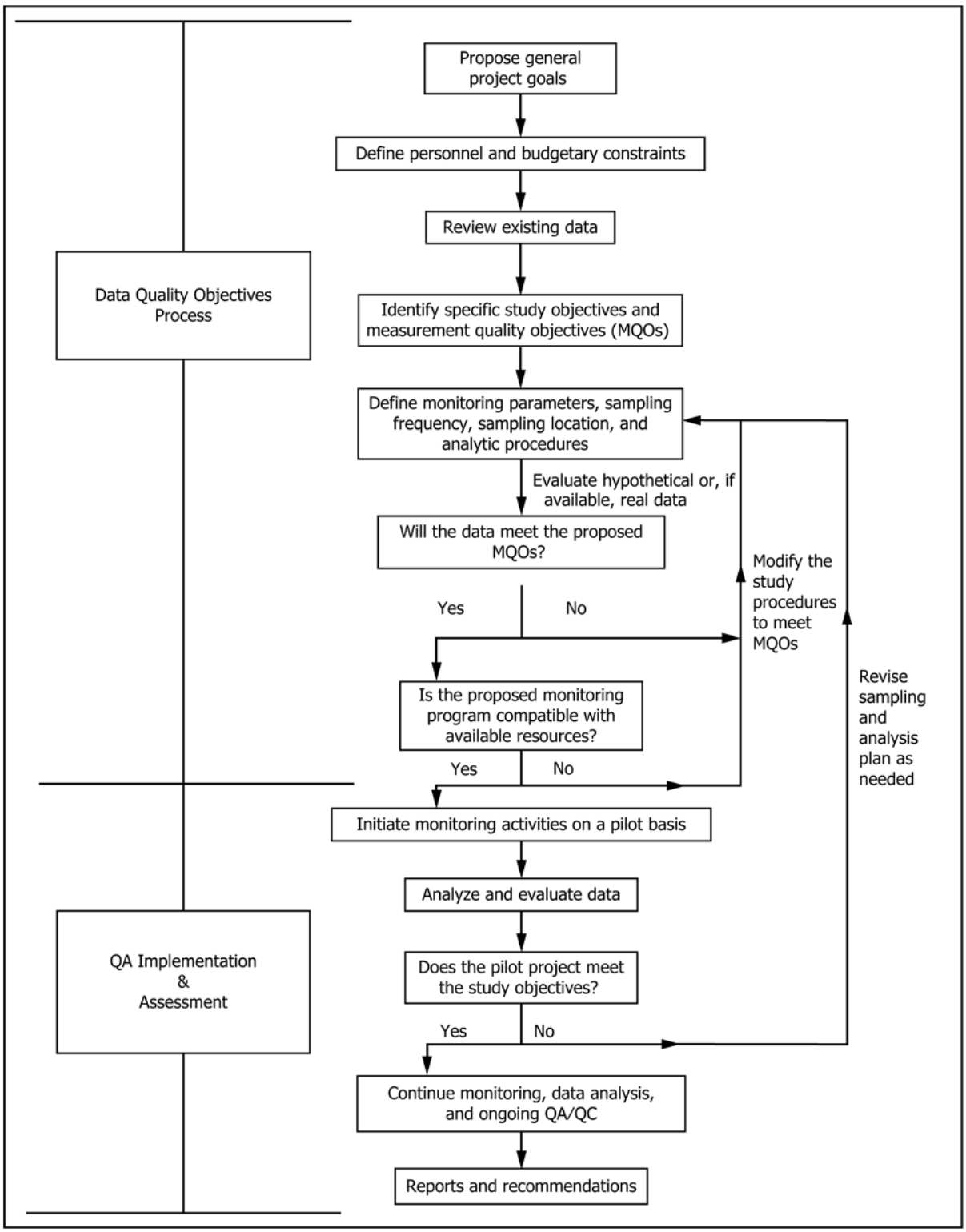

FIG. 1 Flow Chart Summarizing the Process that Should Be Implemented in Designing and Performing a Monitoring Study (modified from MacDonald et al. (1991 (10)); USEPA 2001 (1))

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meet the project objectives (for example, specific parameters to be measured) and support a decision based on the results of data collection and observation. Not doing so creates the risk of expending too much effort on data collection (that is, more data are collected than necessary), not expending enough effort on data collection (that is, more data are necessary than were collected), or expending the wrong effort (that is, the wrong data are collected).

9.3 *Data Quality Objectives Process:*

9.3.1 The Data Quality Objectives (DQO) Process developed by USEPA (GLNPO, 1994 (**11**); USEPA, 2000a(**12**)) is a flexible planning tool that systematically addresses the above issues in a coherent manner. The purpose of this process is to improve the effectiveness, efficiency, and defensibility of decisions made based on the data collected, and to do so in an effective manner (USEPA, 2000a(**12**)). The information compiled in the DQO process is used to develop a project-specific Quality Assurance Project Plan (QAPP; Section 10, USEPA 2000a (**12**)) that should be used to plan the majority of sediment quality monitoring or assessment studies. In some instances, a QAPP may be prepared, as necessary, on a project-by-project basis.

9.3.2 The DQO process addresses the uses of the data (most importantly, the decision(s) to be made) and other factors that will influence the type and amount of data to be collected (for example, the problem being addressed, existing information, information needed before a decision can be made, and available resources). From these factors the qualitative and quantitative data needs are determined Fig. 2. DQOs are qualitative and quantitative statements that clarify the purpose

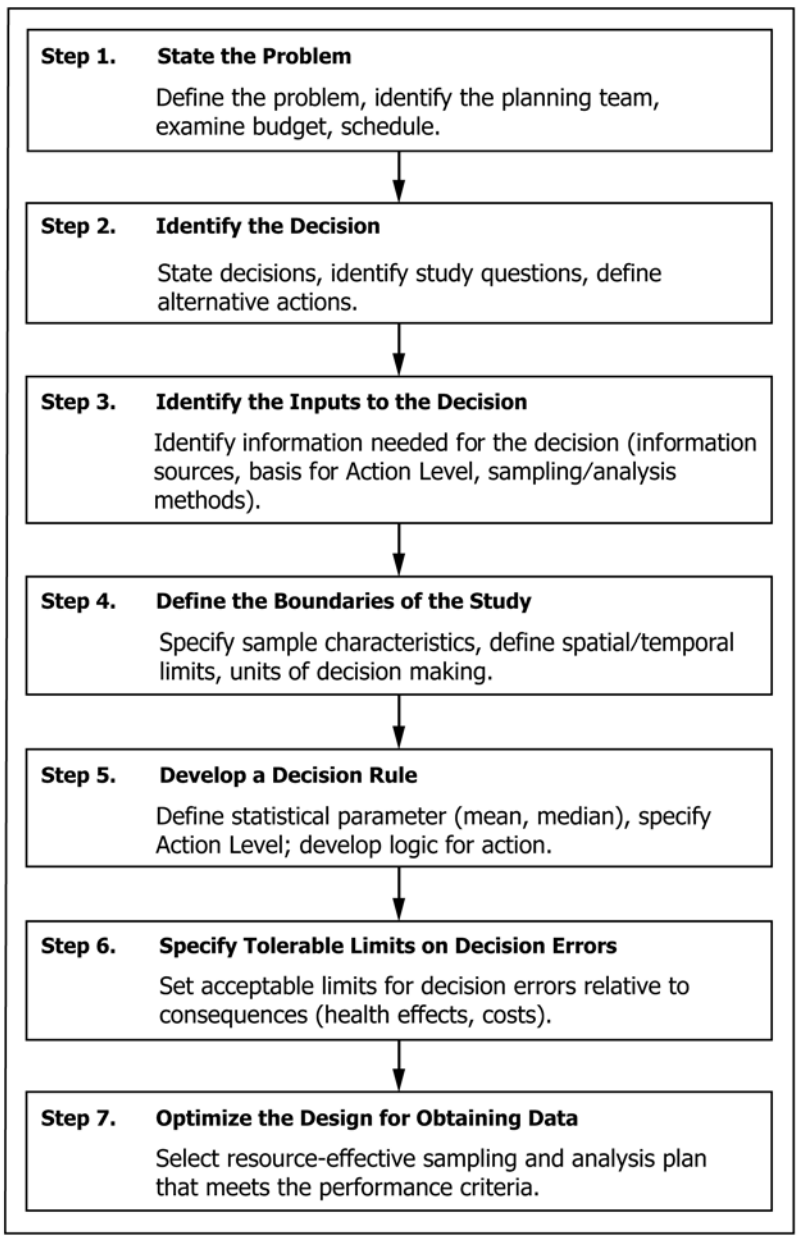

FIG. 2 Flow Chart Summarizing the Data Quality Objectives Process (after USEPA 2000a (12); 2001(10))

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of the monitoring study, define the most appropriate type of data to collect, and determine the most appropriate methods and conditions under which to collect them. The products of the DQO process are criteria for data quality, and a data collection design to ensure that data will meet the criteria.

9.3.3 For most instances, a Sampling and Analysis Plan (SAP) is developed before sampling that describes the study objectives, sampling design and procedures, and other aspects of the DQO process outlined above (USEPA 2001(**1**)). The following sections provide guidance on many of the primary issues that should be addressed in a study plan.

9.4 *Study Plan Considerations:*

9.4.1 *Definition of the Study Area and Study Site:*

9.4.1.1 Monitoring and assessment studies are performed for a variety of reasons (ITFM, 1995 (**13**)) and sediment assessment studies can serve many different purposes. Developing an appropriate sampling plan is one of the most important steps in monitoring and assessment studies. The sampling plan, including definition of the site (a study area that can be comprised of multiple sampling stations) and sampling design, will be a product of the general study objectives Fig. 1. Station location, selection, and sampling methods will necessarily follow from the study design. Ultimately, the study plan should control extraneous sources of variability or error to the extent possible so that data are appropriately representative of the sediment quality, and fulfill the study objectives.

9.4.1.2 The study area refers to the body of water that contains the study sampling stations(s) to be monitored or assessed, as well as adjacent areas (land or water) that might affect or influence the conditions of the study site. The study site refers to the body of water and associated sediments to be monitored or assessed.

9.4.1.3 The size of the study area will influence the type of sampling design (see 9.5) and site positioning methods that are appropriate (see 9.8). The boundaries of the study area need to be clearly defined at the outset and should be outlined on a hydrographic chart or topographic map.

9.4.2 *Controlling Sources of Variability:*

9.4.2.1 A key factor in effectively designing a sediment quality study is controlling those sources of variability in which one is not interested (USEPA 2000a,b (**12**),(**14**)). There are two major sources of variability that, with proper planning, can be minimized, or at least accounted for, in the design process. In statistical terms, the two sources of variability are sampling error and measurement error (USEPA 2000b(**14**); Solomon et al. 1997 (**15**)).

9.4.2.2 Sampling error is the error attributable to selecting a certain sampling station that might not be representative of the site or population of sample units. Sampling error is controlled by either: (*1*) using unbiased methods to select stations if one is performing general monitoring of a given site (USEPA, 2000b (**14**)) or (*2*) selecting several stations along a spatial gradient if a specific location is being targeted (see 9.5).

9.4.2.3 Measurement error is the degree to which the investigator accurately characterizes the sampling unit or station. Thus, measurement error includes components of natural spatial and temporal variability within the sample unit as well as actual errors of omission or commission by the investigator. Measurement error is controlled by using consistent and comparable methods. To help minimize measurement error, each station should be sampled in the same way within a site, using a consistent set of procedures and in the same time frame to minimize confounding sources of variability (see 9.4.3). In analytical laboratory or toxicity procedures, measurement error is estimated by duplicate determinations on some subset of samples (but not necessarily all). Similarly, in field investigations, some subset of sample units (for example, 10 % of the stations) should be measured more than once to estimate measurement error (see Replicate and Composite Samples, 9.6.7). Measurement error can be reduced by analyzing multiple observations at each station (for example, multiple grab samples at each sampling station, multiple observations during a season), or by collecting depth-integrated, or spatially integrated (composite) samples (see 9.6.7).

9.4.2.4 Optimizing the sampling design requires consideration of tradeoffs among the procedures used to analyze data. These include, the effect that is considered meaningful, desired power, desired confidence, and resources available for the sampling program (Test Method E1706). Most studies do not estimate power of their sampling design because this generally requires prior information such as pilot sampling, which entails further resources. One study (Gilfillan et al. 1995 (**16**)) reported power estimates for a shoreline monitoring program following the Valdez oil spill in Prince William Sound, Alaska. However, these estimates were computed after the sampling took place. It is desirable to estimate power before sampling is performed to evaluate the credibility of non-significant results (see for example, Appendix C in USEPA 2001(**1**)).

9.4.2.5 Measures of bioaccumulation from sediments depend on the exposure of the organism to the sample selected to represent the sediment concentration of interest. It is important to match as close as possible the sample selected for measuring the sediment chemistry to the biology of the organism (Lee 1991(**17**), Test Method E1706). For instance, if the organism is a surface deposit feeder, the sediment sample should to the extent possible represent the surficial feeding zone of the organism. Likewise if the organism feeds at depth, the sediment sample should represent that feeding zone.

9.4.3 *Sampling Using an Index Period:*

9.4.3.1 Most monitoring projects do not have the resources to characterize variability or to assess sediment quality for all seasons. Sampling can be restricted to an index period when biological or toxicological measures are expected to show the greatest response to contamination stress and within-season variability is small (Holland, 1985 (**18**); Barbour et al. 1999 (**19**)). This type of sampling might be especially advantageous for characterizing sediment toxicity, sediment chemistry, and benthic macroinvertebrate and other biological assemblages (USEPA, 2000c (**20**)). In addition, this approach is useful if sediment contamination is related to, or being separated from, high flow events or if influenced by tidal cycles. By sampling overlying waters during both low and high flow conditions or tidal cycles, the relative contribution of each to contaminant can be better assessed, thereby better directing remedial activities, or other watershed improvements.

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9.4.3.2 Projects that sample the same station over multiple years are interested in obtaining comparable data with which they can assess changes over time, or following remediation (GLNPO, 1994 (**11**)). In these cases, index period sampling is especially useful because hydrological regime (and therefore biological processes) is likely to be more similar between similar seasons than among different seasons.

9.5 *Sampling Designs:*

9.5.1 As mentioned in earlier sections, the type of sampling design used is a function of the study DQOs and more specifically, the types of questions to be answered by the study. A summary of various sampling designs is presented in Fig. 3. Generally, sampling designs fall into two major categories: random (or probabilistic) and targeted (USEPA, 2000b (**14**)). USEPA (2000b,c (**14**),(**20**)) Gilbert (1987 (**21**)), and Wolfe et al. (1993 (**22**)) present discussions of sampling design issues and information on different sampling designs. Appendix A in USEPA (2001, (**1**)) presents hypothetical examples of sediment quality monitoring designs given different objectives or regulatory applications.

9.5.2 *Probabilistic and Random Sampling:*

9.5.2.1 Probability-based or random sampling designs avoid bias in the sample results by randomly assigning and selecting sampling locations. A probability design requires that all sampling units have a known probability of being selected. Both the USPEA Environmental Monitoring Assessment Program and the NOAA National Status and Trends Program use a probabilistic sampling design to infer regional and national patterns with respect to contamination or biological effects.

9.5.2.2 Stations can be selected on the basis of a truly random scheme or in a systematic way (for example, sample every 10 m along a randomly chosen transect). In simple random sampling, all sampling units have an equal probability of selection. This design is appropriate for estimating means and totals of environmental variables if the population is homogeneous. To apply simple random sampling, it is necessary to identify all potential sampling times or locations, then randomly select individual times or locations for sampling.

9.5.2.3 In grid or systematic sampling, the first sampling location is chosen randomly and all subsequent stations are placed at regular intervals (for example, 50 m apart) throughout the study area. Clearly, the number of sampling locations could be large if the study area is large and one desires "fine-grained" contaminant or toxicological information. Thus, depending on the types of analyses desired, such sampling might become expensive unless the study area is relatively small, or the density of stations (that is, how closely spaced are the stations) is relatively low. Grid sampling might be effective for detecting previously unknown "hot spots" in a limited study area.

9.5.2.4 In stratified designs, the selection probabilities might differ among strata. Stratified random sampling consists of dividing the target population into non-overlapping parts or subregions (for example, ecoregions, watersheds, or specific dredging or remediation sites) termed strata to obtain a better estimate of the mean or total for the entire population. The information required to delineate the strata and to estimate sampling frequency should either be known before sampling

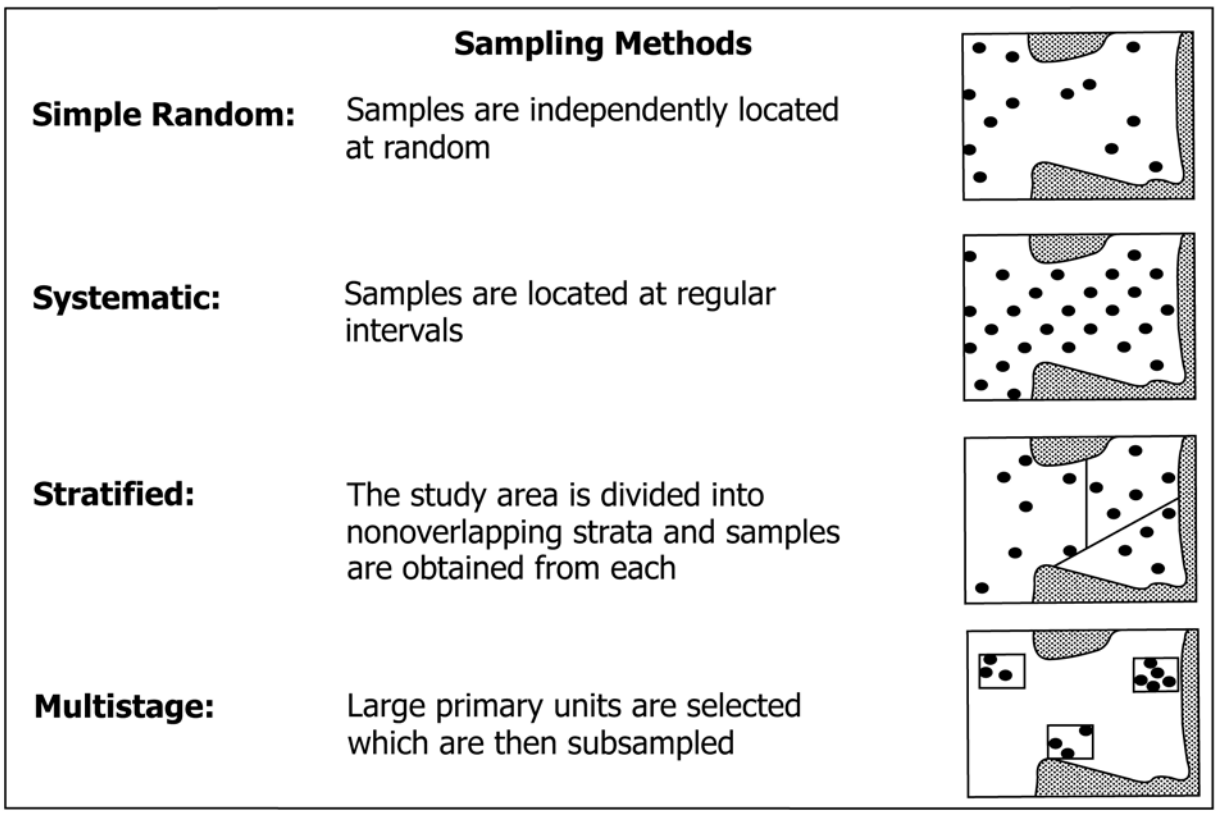

FIG. 3 Description of Various Sampling Methods (adapted from USEPA 2000c (20); 2001(1))

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using historic data variability, available information and knowledge of ecological function, or obtained in a pilot study. Sampling locations are randomly selected from within each of the strata. Stratified random sampling is often used in sediment quality monitoring because certain environmental variables can vary by time of day, season, hydrodynamics, or other factors. One disadvantage of using random designs is the possibility of encountering unsampleable stations that were randomly selected by the computer. Such problems result in the need to reposition the vessel to an alternate location (Heimbuch et al. 1995 (**23**), Strobel et al. 1995 (**24**)) Furthermore, if one is sampling to determine the percent spatial extent of degradation, it might be important to sample beyond the boundaries of the study area to better evaluate the limits of the impacted area.

9.5.2.5 A related design is multistage sampling in which large subareas within the study area are first selected (usually on the basis of professional knowledge or previously collected information). Stations are then randomly located within each subarea to yield average or pooled estimates of the variables of interest (for example, concentration of a particular contaminant or acute toxicity to the amphipod Hyalella azteca) for each subarea. This type of sampling is especially useful for statistically comparing variables among specific parts of a study area.

9.5.2.6 Use of random sampling designs might also miss relationships among variables, especially if there is a relationship between an explanatory and a response variable. As an example, estimation of benthic response or contaminant concentration, in relation to a discharge or landfill leachate stream, requires sampling targeted locations or stations around the potential contaminant source, including stations presumably unaffected by the source (for example, Warwick and Clarke, 1991(**25**)). A simple random selection of stations is not likely to capture the entire range needed because most stations would likely be relatively removed from the location of interest.

9.5.3 *Targeted Sampling Designs:*

9.5.3.1 In targeted (also referred to as judgmental, or model-based) designs, stations are selected based on prior knowledge of other factors, such as salinity, substrate type, and construction or engineering considerations (for example, dredging). The sediment studies conducted in the Clark Fork River (Pascoe and DalSoglio, 1994 (**26**); Brumbaugh et al. 1994 (**27**)), in which contaminated areas were a focus, used a targeted sampling design.

9.5.3.2 Targeted designs are useful if the objective of the investigation is to screen an area(s) for the presence or absence of contamination at levels of concern, such as risk-based screening levels, or to compare specific sediment quality against reference conditions or biological guidelines. In general, targeted sampling is appropriate for situations in which any of the following apply (USEPA, 2000b (**14**)):

(1) The site boundaries are well defined or the site physically distinct (for example, USEPA Superfund or CERCLA site, proposed dredging unit).

(2) Small numbers of samples will be selected for analysis or characterization.

(3) Information is desired for a particular condition (for example, "worst case") or location.

(4) There is reliable historical and physical knowledge about the feature or condition under investigation.

(5) The objective of the investigation is to screen an area(s) for the presence or absence of contamination at levels of concern, such as risk-based screening levels. If such contamination is found, follow-up sampling is likely to involve one or more statistical designs to compare specific sediment quality against reference conditions.

(6) Schedule or budget limitations preclude the possibility of implementing a statistical design.

(7) Experimental testing of a known contaminant gradient to develop or verify testing methods or models (that is, as in evaluations of toxicity tests, Long et al. 1990 (**28**)).

9.5.3.3 Because targeted sampling designs often can be quickly implemented at a relatively low cost, this type of sampling can often meet schedule and budgetary constraints that cannot be met by implementing a statistical design. In many situations, targeted sampling offers an additional important benefit of providing an appropriate level-of-effort for meeting investigation objectives without excessive use of project resources.

9.5.3.4 Targeted sampling, however, limits the inferences made to the stations actually sampled and analyzed. Extrapolation from those stations to the overall population from which the stations were sampled is subject to unknown selection bias. This bias might be unimportant for programs in which information is needed for a particular condition or location).

9.6 *Measurement Quality Objectives:*

9.6.1 As noted in 9.3, a key aspect of the DQO process is specifying measurement quality objectives (MQOs): statements that describe the amount, type, and quality of data needed to address the overall project objectives Table 1.

9.6.2 A key factor determining the types of MQOs needed in a given project or study is the types of analyses required because these will determine the amount of sample required (see 9.6.5) and how samples are processed (see Section11). Metals, organic chemicals (including pesticides, PAHs, and PCBs), whole sediment toxicity, and organism bioaccumulation of specific target chemicals, are frequently analyzed in many sediment monitoring programs.

9.6.3 A number of other, more "conventional" parameters, are also often analyzed as well to help interpret chemical, biological, and toxicological data collected in a project (see Section 14). Table 2 summarizes many of the commonly measured conventional parameters and their uses in sediment quality studies (WDE, 1995 (**29**)). It is important that conventional parameters receive as much careful attention, in terms of sampling and sample processing procedures, as do the contaminants or parameters of direct interest. The guidance presented in Sections 10 and 11 provides information on proper sampling and sample processing procedures to establish that one has appropriate samples for these analyses.

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TABLE 1 Checklist for the DQO Process (USEPA 2001(1))

Clearly state the problem: purpose and objectives, available resources, members of the project team: For example, the purpose might be to evaluate current sediment quality conditions, historical conditions, evaluate remediation effects, or validate a sediment model. It is important to review and evaluate available historical data relevant to the study at this point in the process.

Identify the decision; the questions(s) the study attempts to address: For example, is site A more toxic than site B?; Are sediments in Lake Y less toxic now than they used to be?; Does the sediment at site D need to be remediated? What point or nonpoint sources are contributing to sediment contamination?

Identify inputs to the decision: information and measurements that need to be obtained: For example, analyses of specific contaminants, toxicity test results, biological assessments, bioaccumulation data, habitat assessments, hydrology, and water quality characterization.

Define the study boundaries (spatial and temporal): Identify potential sources of contamination; determine the location of sediment deposition zones; determine the frequency of sampling and need for a seasonal sampling and/or sampling during a specific index period; consider areas of previous dredged or fill material discharges/disposal. Consideration of hydraulic patterns, flow event frequency, and/or sedimentation rates could be critical for determining sampling frequency and locations.

Develop a decision rule: define parameters of interest and determine the value of a parameter that would cause follow-up action of some kind: For example., exceedance of Sediment Quality Guidelines (Wenning and Ingersoll 2002 (6)) or toxicity effect results in some action. For example, in the Great Lakes Assessment and Remediation of Contaminated Sediments (ARCS) Program, one decision rule was: if total PCB concentration exceeds a particular action level, then the sediments will be classified as toxic and considered for remediation (GLNPO, 1994 (11)).

Specify limits on decision errors: Establish the measurement quality objectives (MQOs) which include determining the level of confidence required from the data; precision, bids, representativeness, and completeness of data; the sample size (weight or volume) required to satisfy the analytical methods and QA/QC program for all analytical tests; the number of samples required, to be within limits on decision errors, and compositing needed, if any.

Optimize the design: Choose appropriate sampling and processing methods; select appropriate method for determining the location of sampling stations; select an appropriate positioning method for the site and study. Consult historical data and a statistician before the study begins regarding the sampling design (i.e., the frequency, number, and location of field-collected samples) that will best satisfy study objectives.

TABLE 2 Conventional Sediment Variables and Their Use in Sediment Investigations (adapted from WDE, 1995(29) and USEPA 2001(1))

Conventional Sediment Variable	Use
Total organic carbon (TOC)	Normalization of the concentrations of nonionizable organic compounds Identification of appropriate reference sediments for biological tests
Acid Volatile Sulfide (AVS)	Normalization of the concentrations of divalent metals in anoxic sediments
Sediment grain size	Identification of appropriate reference sediments for biological tests Interpretation of sediment toxicity test data and benthic macroinvertebrate abundance data Evaluation of sediment transport and deposition Evaluation of remedial alternatives
Total solids	Expression of chemical concentrations on a dry-weight basis
Ammonia	Interpretation of sediment toxicity test data
Total sulfides	Interpretation of sediment toxicity test data

TABLE 3 Typical Sediment Volume Requirements for Various Analyses per Sample (USEPA 2001(1))

Sediment Analysis	Minimum Sample Volume
Inorganic chemicals	90 mL
Non-petroleum organic chemicals	230 mL
Other chemical parameters (for example, total organic carbon, moisture content)	300 mL
Particle size	230 mL
Petroleum hydrocarbons[A]	250 to 1000 mL
Acute and chronic whole sediment toxicity tests[B]	1 to 2 L
Bioaccumulation tests[C]	15 L
Benthic macroinvertebrate assessments	8 to 16 L
Pore water extraction	2 L
Elutriate preparation	1 L

[A] The maximum volume (1000 mL) is required only for oil and grease analysis; otherwise, 250 mL is sufficient.
[B] Amount needed per whole sediment test (that is, one species) assuming 8 replicates per sample and test volumes specified in USEPA, 2000d(30).
[C] Based on an average of 3 L of sediment per test chamber and 5 replicates (USEPA, 2000d(30)).

9.6.4 The following sections concentrate on three aspects of MQO development that are generally applicable to all sediment quality studies, regardless of the particular objectives: sample volume, number of samples, and replication versus composite sampling.

9.6.5 *Sample Volume:*

9.6.5.1 Before commencing a sampling program, the type and number of analyses and tests should be determined, and the required volume of sediment per sample calculated. Each physicochemical and biological test requires a specific amount of sediment which, for chemical analyses, depends on the detection limits attainable and extraction efficiency by the analytical procedure and, for biological testing, depends on the test organisms and method. Typical sediment volume requirements for each end use are summarized in Table 3. Recommendations for determining the number of samples and sample volume are presented in Table 4.

9.6.5.2 When determining the required sample volume, it is important to know all of the required sample analyses (considering adequate replication), and it is also useful to know the general characteristics of the sediments being sampled. For example, if interstitial water analyses or elutriate tests are to be conducted, the percent water (or percent dry weight) of the sediment will greatly affect the amount of water extracted. Many non-compacted, depositional sediments have interstitial water contents often ranging from 30 to 70 %. However, there is a low volume of water in these types of sediments.

9.6.5.3 For benthic macroinvertebrate bioassessment analyses, sampling a prescribed area of benthic substrate is at least as important as sampling a given volume of sediment (Annex A1). Macroinvertebrates are often sampled using multiple grab samples within a given station location, typically to a consistent sediment depth (for example, per 100 cm of

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TABLE 4 Recommendations on Determining How Many Samples and How Much Sample Volume Should Be Collected (USEPA 2001(1))

The testing laboratory should be consulted to confirm the amount of sediment required for all desired analyses.
The amount of sediment needed from a given site will depend on the number and types of analyses to be performed. If biological, toxicological, and chemical analyses are required (sediment triad approach), then at least 10 L of sediment might be required from each station.
Since sampling events might be expensive and/or difficult to replicate, it is useful to collect extra samples if possible, in the event of problems encountered by the analytical laboratories, failure of performance criteria in assays, or need to verify/validate results.
Consider compositing samples from a given station or across similar station types to reduce the number of samples needed.

sediment; Klemm et al. 1990 (**31**); GLNPO, 1994 (**11**); Long et al. 1996 (**32**); USEPA 2000c (**20**)). More than 6 liters of sediment from each station might be necessary in order to have adequate numbers of organisms for analyses, especially in many lakes, estuaries, and large rivers (Barbour et al. 1999 (**19**)). However, this is very site specific, and should be determined by the field sampling crew. This only applies to whole sediment sampling methods and not to surficial stream methods using methods such as kick-nets and Surber samplers. If the sediment quality triad approach is used (that is, biological, toxicological, and physicochemical analyses performed on samples from the same stations), more than 10 liters of sediment from each station might be required depending on the specific analyses conducted. NOAA routinely collects 7 to 8 liters of sediment at each station for multiple toxicity tests and chemical analyses (Long et al. 1996 (**32**)).

9.6.6 *Number of Samples:*

9.6.6.1 The number of samples collected directly affects the representativeness and completeness of the data for purposes of addressing project goals Table 4. As a general rule, a greater number of samples will yield better definition of the areal extent of contamination or toxicity.

9.6.6.2 Accordingly, sample requirements should be determined on a case-by-case basis. The number of samples to be collected will ultimately be an outcome of the questions asked. For example, if one is interested in characterizing effects of a point source or a gradient (for example, effects of certain tributaries or land uses on a lake or estuary), then many samples in a relatively small area might need to be collected and analyzed. If, however, one is interested in screening "hot spots" or locations of high contamination within a watershed or water body, relatively few samples at regularly-spaced locations might be appropriate. In most monitoring and assessment studies, the number of samples to be collected usually results from a compromise between the ideal and the practical. The major practical constraints are the costs of analyses and logistics of sample collection.

9.6.6.3 The major costs associated with the collection of sediment samples are those for travel to the site and for sample analysis. The costs of actual on-site sampling are minimal by comparison. Consequently, it is good practice to collect an excess number of samples, and then a subset equal to the minimum number required is selected for analysis. The archived replicate samples can be used to replace lost samples, for data verification, to rerun analyses yielding questionable results, or for the independent testing of a posteriori hypotheses that might arise from screening the initial data. However, storage of sediments might result in changes in bioavailability of chemical contaminants (see 11.6) or in exceeding analytical holding times. Therefore, follow-up testing of archived samples should be done cautiously.

9.6.7 *Replicate and Composite Samples:*

9.6.7.1 *Replicate samples*: As mentioned in the previous section, the number of samples collected and analyzed will always be a compromise between the desire of obtaining high quality data that fully addresses the overall project objectives (MQOs), and the constraints imposed by analytical costs, sampling effort, and study logistics. Therefore, each study needs to find a balance between obtaining information to satisfy the stated DQOs or study goals in a cost-effective manner, and yet have enough confidence in the data to make appropriate decisions (for example, remediation, dredging; Step 3 in the DQO process, Fig. 2). Two different concepts are used to satisfy this challenge: replication and sample compositing.

9.6.7.2 Replication is used to assess precision of a particular measure and can take many forms depending on the type of precision desired. For most studies, analytical replicates are the most frequently used form of replication because most MQOs are concerned with analytical data quality (USEPA 2001(**1**)). The extent of analytical replication (duplicates) varies with the study DQOs. Performing duplicate analyses on at least 10 % of the samples collected is considered satisfactory for most studies (GLNPO, 1994 (**11**); USEPA/USACE, 1991(**33**); PSEP, 1997a (**34**); USEPA/USACE, 1998 (**35**)). An MQO of less than 20 to 30 % relative percent difference (RPD) is commonly used for analytical replicates depending on the analyte.

9.6.7.3 Field replicates can provide useful information on the spatial distribution of contaminants at a station and the heterogeneity of sediment quality within a site. Furthermore, field replicates provide true replication at a station (analytical replicates and split samples at a station provide a measure of precision for a given sample, not the station) and therefore can be used to statistically compare analyses (for example, toxicity, tissue concentration, whole sediment concentration) across stations.

9.6.7.4 Results of field replicate analysis yield the overall variability or precision of both the field and laboratory operations (as well as the variability between the replicate samples themselves, apart from any procedural error). Because field replicate analyses integrate a number of different sources of variability, they might be difficult to interpret. As a result, failure to meet a precision MQO for field replicates might or might not be a cause of concern in terms of the overall study objectives, but would suggest some uncertainty in the data. Many monitoring programs perform field replicates at 10 % of the stations sampled in the study as a quality control procedure. An MQO of less than 30 to 50 % relative percent difference (RPD) is typically used for field replicates depending on the analyte (USEPA 2001(**1**)). Many regulatory programs (for

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example, Dredged Disposal Management within the Puget Sound Estuary Program) routinely use 3 to 5 field replicates per station. Appendix C of USEPA (2001 (**1**)) summarizes statistical considerations in determining the appropriate number of replicate samples given different sampling objectives.

9.6.7.5 Split sample replication is less commonly performed in the field because many investigators find it more useful to quantify data precision through the use of analytical and field replicates described above. However, split sample replication is frequently used in the laboratory in toxicity and bioaccumulation analyses (USEPA, 2000d (**30**)) and to verify homogeneity of test material in spiked sediment tests (see 12.4). In the field, samples are commonly split for different types of analyses (for example, toxicity, chemistry, benthos) or for inter-laboratory comparisons rather than to replicate a given sample. This type of sample splitting or subsampling is further discussed in 11.3.

9.6.7.6 *Composite Samples*—A composite sample is one that is formed by combining material from more than one sample or subsample. Because a composite sample is a combination of individual aliquots, it represents an "average" of the characteristics making up the sample. Compositing, therefore, results in a less detailed description of the variability within the site as compared to taking field replicates at each station. However, for characterizing a single station, compositing is generally considered a good way to provide quality data with relatively low uncertainty. Furthermore, many investigators find it useful to average the naturally heterogeneous physicochemical conditions that often exist within a station (or dredging unit, for example), even within a relatively small area (GLNPO, 1994 (**11**); PSEP, 1997a(**34**)). Some investigations have composited 3 to 5 samples from a given location or depth strata (GLNPO, 1994 (**11**)).

9.6.7.7 Compositing is also a practical way to control analytical costs while providing information from a large number of stations. For example, with relatively little more sampling effort, five analyses can be performed to characterize a project segment or site by collecting 15 samples and combining sets of three into five composite samples. The increased coverage afforded by taking composite samples might justify the increased time and cost of collecting the extra 10 samples in this case (USEPA/USACE, 1998 (**35**)). Compositing is also an important way to provide the large sample volumes required for some biological tests and for multiple types of analyses (for example, physical, chemical, toxicity, and benthos). However, compositing is not recommended where combining samples could serve to "dilute" a highly toxic but localized sediment "hot spot" (WDE, 1995 (**29**); USEPA/USACE, 1998 (**35**)). Also, samples from stations with very different grain size characteristics or different stratigraphic layers of core samples should not be composited (see 11.4).

9.7 *Site-Specific Considerations for Selecting Sediment Sampling Stations:*

9.7.1 Several site-specific factors might ultimately influence the appropriate location of sampling stations, both for large-scale monitoring studies, in which general sediment quality status is desired, and for smaller, targeted studies. If a targeted or stratified random sampling design is chosen, it might be important to locate sediment depositional and erosional areas to properly identify contaminant distributions. Tables 5 and 6 presents a summary of site-specific factors that should be considered when developing a sampling plan. A more detailed review of such considerations is provided by Mudroch and MacKnight (1994 (**36**)).

9.7.2 *Review Available Data*—Review of available historical and physical data is important in the sample selection process and subsequent data interpretation. Local experts should be consulted to obtain information on site conditions and the origin, nature, and degree of contamination. Other potential sources of information include government agency records, municipal archives, harbor commission records, past geochemical analyses, hydrographic surveys, bathymetric maps, and dredging or disposal history. Potential sources of contamination should be identified and their locations noted on a map or chart of the proposed study area. It is important that recent hydrographic or bathymetric data be used in identifying

TABLE 5 Practical Considerations for Selection of Sampling Stations in Developing a Sampling Plan (USEPA 2001(1))

Activity	Consideration
Determination of areas where sediment contamination might occur	Hydrologic information: quality and quantity of runoff potential depositional inputs of total suspended solids up-wellings seepage patterns
Determination of depositional and erosional areas	Bathymetric maps and hydrographic charts: water depth zones of erosion, transport, and deposition bathymetry distribution, thickness, and type of sediment velocity and direction of currents sedimentation rates Climatic conditions: prevailing winds seasonal changes in temperature, precipitation, solar radiation, etc. tides, seiches seasonal changes in anthropogenic and natural loadings
Determination of potential sources of contamination	Anthropogenic considerations: location of urban lefts historical changes in land use types, densities, and size of industries location of waste disposal sites location of sewage treatment facilities location of stormwater outfalls and combined sewer overflows location, quantity, and quality of effluents previous monitoring and assessment or geochemical surveys location of dredging and open-water dredged material disposal sites location of historical waste spills
Factors affecting contaminant bioavailability	Geochemical considerations: type of bedrock and soil/sediment chemistry physical and chemical properties of overlying water
Determination of representativeness of samples	area to be characterized volume to be characterized depth to be characterized possible stratification of the deposit to be characterized

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TABLE 6 Recommendations for Positioning of Sampling Stations (USEPA 2001 (1))

Depending on level of accuracy needed, regular calibration of the positioning system by at least two methods might be required to ensure accuracy.
For monitoring and assessment studies of large areas (for example, large lakes or offshore marine environments), where an accuracy of ± 100 m typically is sufficient, either the Long Range Navigation (LORAN) or Global Positioning System (GPS) system is recommended.
For near-shore areas, or areas where the sampling stations are numerous or located relatively close together, GPS or a microwave system should be used if the required position accuracy is less than 10 m. Where visible or suitable and permanent targets are available, RADAR can be used if the required position accuracy is between 10 and 100 m.
For small water bodies and urban waterfronts, GPS is often capable of giving precise location information. Alternatively, visual angular measurements (for example, sextant) by an experienced operator, a distance line, or taut wire could also provide accurate and precise positioning data.

representative sampling locations, especially for dredging or other sediment removal projects. The map or chart should also note adjacent land and water uses (for example, fuel docks, storm drains). The quality and age of the available data should be considered, as well as the variability of the data.

9.7.3 *Site Inspection:*

9.7.3.1 A physical inspection of the site should be performed when developing a study plan in order to assess the completeness and validity of the collected historical data, and to identify any significant changes that might have occurred at the site or study area (Mudroch and MacKnight, 1994 (**36**)). A site inspection of the immediate drainage area and upstream watershed might also identify potential stressors (such as erosion), and help determine appropriate sampling gear (such as corer vs. grab samplers and boat type), and sampling logistics.

9.7.3.2 If resources allow, it is useful to perform some screening or pilot sampling and analyses at this stage to further refine the actual sampling design needed. Pilot sampling is particularly helpful in defining appropriate station locations for targeted sampling, or to identify appropriate strata or subareas in stratified or multistage sampling.

9.7.4 *Identify Sediment Deposition and Erosional Zones:*

9.7.4.1 When study DQOs target sampling to the highest contamination levels or specific subareas of a site, it might be important to consider sediment deposition and sediment erosional zones, since grain size and related physicochemical characteristics (including conventional parameters, such as total organic carbon and acid volatile sulfide, as well as other contaminants), are likely to vary between these two types of zones. Depositional zones typically contain fine-grained sediment deposits which are targeted in some sampling programs because fine-grained sediments tend to have higher organic carbon content (and are therefore a more likely repository for contaminants) relative to larger sediment particle size fractions (for example, sand and gravel; Environment Canada 1994(**2**), USEPA 2001(**1**)). However, for some studies such as remediation dredging evaluations or USEPA Superfund sites, eroding sediment beds and non-depositional zones might be of most concern as these could be a major source of contaminants in the water column and in organisms USEPA/USACE,(1991 (**33**)).

9.7.4.2 Various non-disruptive technologies are available to assist in the location of fine-grained sediments ranging from simplistic to more advanced. For example, use of a steel rod or PVC pipe can be used in many shallow areas to quickly and easily probe the sediment surface to find coarse (sand, gravel) vs. fine sediments (silt, clay). This technique can not, however, determine sediment grain size at depth. Other more advance methods, including acoustic survey techniques (for example, low frequency echo sounding, seismic reflections) and side-scan sonar used with a sub-bottom profiler (Wright et al. 1987 (**37**)), can provide useful information on surficial as well as deeper sediment profiles. However, these techniques are often limited in their accuracy and have high equipment costs (Guignè et al. 1991 (**38**)). Sediment Profile Imaging (SPI) or REMOTS can also assist in the identification of grain size and substrate type in advance of field-sampling activities (Germano 1989 (**39**); Rhoads and Germano 1982 (**40**), 1986 (**41**)).

9.7.4.3 Aerial reconnaissance, with or without satellite imagery, might assist in visually identifying depositional zones where clear water conditions exist. However, these methods are not reliable if the water is turbid. Other methods that can be used to locate sediment deposition zones include grab sampling, inspection by divers, or photography using an underwater television camera or remotely operated vehicle (Burton, 1992 (**42**)).

9.8 *Positioning Methods for Locating Sampling Stations:*

9.8.1 The most important function of positioning technology is to determine the location of the sampling station (for example, latitude and longitude), so that the user can later re-sample to the same position (USEPA, 1987 (**43**)). Knowing the precise location of sampling stations is also important to determine if the area(s) of interest have been sampled. There are a variety of navigation or position-fixing systems available, including optical or line-of-site techniques, electronic positioning systems, and satellite positioning systems. Global Positioning System (GPS) is generally regarded as the positioning technique of choice as it is accurate, readily available, and often less expensive than many other comparably sophisticated systems. Given the removal of selective availability of satellite data by the U.S. military, GPS is now capable of high accuracy positioning (1 to 10 m).

9.8.2 Regardless of the type of system selected, calibration of the system should be done using at least two of these methods to determine accuracy, particularly for stations that may be resampled. At each sampling station, a fathometer or meter wheel can be used to determine the sampling depth. This will help to establish that the water is the desired depth and the bottom is sufficiently horizontal for proper operation of sampling equipment. Ideally, it is best to print out a copy of the ship's location from the GPS monitor navigation chart, as well as the latitude and longitude, so the sampling station can be placed in a spatial context. Tidal or subsurface currents may push either the vessel or its suspended sampler away from the intended location which can lead to inaccurate sampling location.

9.9 *Preparations for Field Sampling*

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9.9.1 Proper preparation for any field sampling study is an essential part of Quality Assurance is important to the successful project outcome and adherence to the objectives specified in the QAPP. Section 15 further discusses related Quality Assurance/Quality Control procedures that should be used in sediment quality studies.

9.9.2 Before performing field work, characteristics of the site and accessibility of the individual sampling stations should be determined. Pictures of sampling stations both before as well as during sampling are often useful to document that the correct stations were sampled, and to document weather and water conditions during sampling. Adequate reconnaissance of stations before sampling is also valuable for preparing against potential sampling hazards or unforeseen difficulties. Such a reconnaissance can also help determine the necessary time needed to perform the desired sampling (that is, time to get from one station to the next).

9.9.3 The appropriate vessel or sampling platform is one of the most important considerations in preparing for field sampling. The vessel should be appropriate for the water body type, and should provide sufficient space and facilities to allow collection, any on-board manipulation, and storage of samples. Ice chests or refrigeration might be required for sample storage, depending on the time course of the operation. The vessel should provide space for storage of decontamination materials, as well as clean sampling gear and containers to minimize contamination associated with normal vessel operations. Space for personal safety equipment is also required.

9.9.4 Additionally, the vessel should be equipped with sufficient winch power and cable strength to handle the weight of the sampling equipment, taking into account the additional suction pressure associated with extraction of the sediments. Large sampling devices typically weigh between 50 and 400 kg empty, and when filled with wet sediment might weigh from 125 to over 500 kg.

9.9.5 Care should be taken in operating the vessel to minimize disturbances of the sediment to be sampled as well as sampling equipment. This would include physical disturbance through propeller action and chemical contamination from engines or stack emissions. For example, Page et al. (1995 a,b (**44**),(**45**)) reported that they positioned the ships' stern into the wind to prevent stack gases from blowing onto sampling equipment during deployment, recovery, and subsampling of sediments in Prince William Sound, Alaska.

9.9.6 The sampling plan and projected time schedule should be posted for view by all personnel. The names, addresses, and telephone numbers of all participants involved with the preparation and execution of the sampling program should be available to all participants, and the duties and responsibilities of each participant clearly documented. The study supervisor should determine that the appropriate personnel clearly understand their role and are capable of carrying out their assigned responsibilities and duties. Contingency planning should address the need for backup personnel in the event of accident or illness.

9.9.7 A variety of sampling and sample handling equipment and supplies are often needed in sediment monitoring studies. Besides the actual samplers themselves (for example, grab or core device to be used), equipment is needed to remove and process the samples such as spatulas, scoops, pans or buckets, and gloves. If it is important to maintain anoxic conditions of the sample, a glove box and inert gas source (for example, nitrogen) is needed. Sample storage and transport equipment and supplies need to be available as well. These include refrigeration, ice chests, dry ice or ice, insulation material to stabilize samples in transport, custody seals, and shipping air bills.

9.9.8 The reagents for cleaning, operating, or calibrating equipment, or for collecting, preserving or processing samples should be handled by appropriately qualified personnel and the appropriate data for health and safety (for example, Material Safety Data Sheets) should be available. Standard operating procedures (including QA/QC requirements) should be readily accessible at all times, to facilitate the proper and safe operation of equipment. Data forms and log books should be prepared in advance so that field notes and data can be quickly and efficiently recorded. Extra forms should be available in the event of a mishap or loss. These forms and books should be waterproof and tear resistant. Under certain circumstances, audio or audio/video recordings might prove valuable.

9.9.9 All equipment used to collect and handle samples should be cleaned and all parts examined to facilitate proper functioning before going into the field. A repair kit should accompany each major piece of equipment in case of equipment failure or loss of removable parts. Backup equipment and sampling gear should be available.

9.9.10 Storage, transport, and sample containers, including extra containers, should be available in the event of loss or breakage (see 11.2 for more information on appropriate containers). These containers should be pre-cleaned and labeled appropriately (that is, with a waterproof adhesive label to which the appropriate data can be added, using an indelible ink pen capable of writing on wet surfaces). The containers should have lids that are fastened securely, and if the samples are collected for legal purposes, they should be transported to and from the field in a locked container with custody seals secured on the lids. Samples to be frozen before analyses should not be filled to the very top of the container. Leave at least 10 % headspace to accommodate expansion during freezing (laying glass jars on their side during freezing may help to reduce the chance of the container breaking during freezing). Whether for legal purposes or not, all samples should be accompanied by a chain-of-custody form that documents field samples to be submitted for analyses (see Section 15). Transport supplies also include shipping air bills and addresses. Whole-sediment sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E1706 and Guide E1688).

9.9.11 A sample-inventory log and a sample-tracking log should be prepared in advance of sampling. A single person should be responsible for these logs who will track the samples from the time they are collected until they are analyzed and disposed of or archived.

10. Collection of Whole Sediment Samples

10.1 *General Procedures:*

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10.1.1 Most sediment collection devices are designed to isolate and retrieve a specified volume and surface area of sediment, from a required depth below the sediment surface, with minimal disruption of the integrity of the sample and no contamination of the sample. Maintaining the integrity of the collected sediment, for the purposes of the measurements intended, is a primary concern in most studies because any disruption of the sediment structure changes its physicochemical and biological characteristics, thereby influencing the bioavailability of contaminants and the potential toxicity of the sediment. This section discusses the factors to be considered in selecting a sediment collection device and minimizing disruption of sediment samples. A variety of samplers are described (Annex A1), and recommendations are made regarding their use in different situations.

10.1.2 Figs. 4 and 5 provide suggested grab and core samplers based on site factors (such as depth and particle size), and sampling requirements (such as sample depth and volume of sample needed).

10.1.3 The planned mode of access to the sampling area (for example, by water, over land or ice, or from the air) plays an important role in the selection of sampling gear. If the sampling gear needs to be transported to a remote area or shipped by air, its weight and volume might should be taken into account. It is often the case that a specific vessel, having a fixed lifting capacity based on the configuration of its winch, crane, boom, A-frame, or other support equipment, is the only one available for use. This will affect the type of sampling equipment that can be safely operated from that vessel.

10.1.4 Many samplers are capable of recovering a relatively undisturbed sample in soft, fine-grained sediments, but fewer are suitable for sampling harder sediments containing significant quantities of sand, gravel, firm clay, or till (Mudroch and Azcue, 1995 (**46**)). One of the most important factors in determining the appropriate sampling device for the study are DQOs. Many monitoring programs, such as the USEPA Environmental Monitoring and Assessment Program (EMAP) and the NOAA National Status and Trends program, are

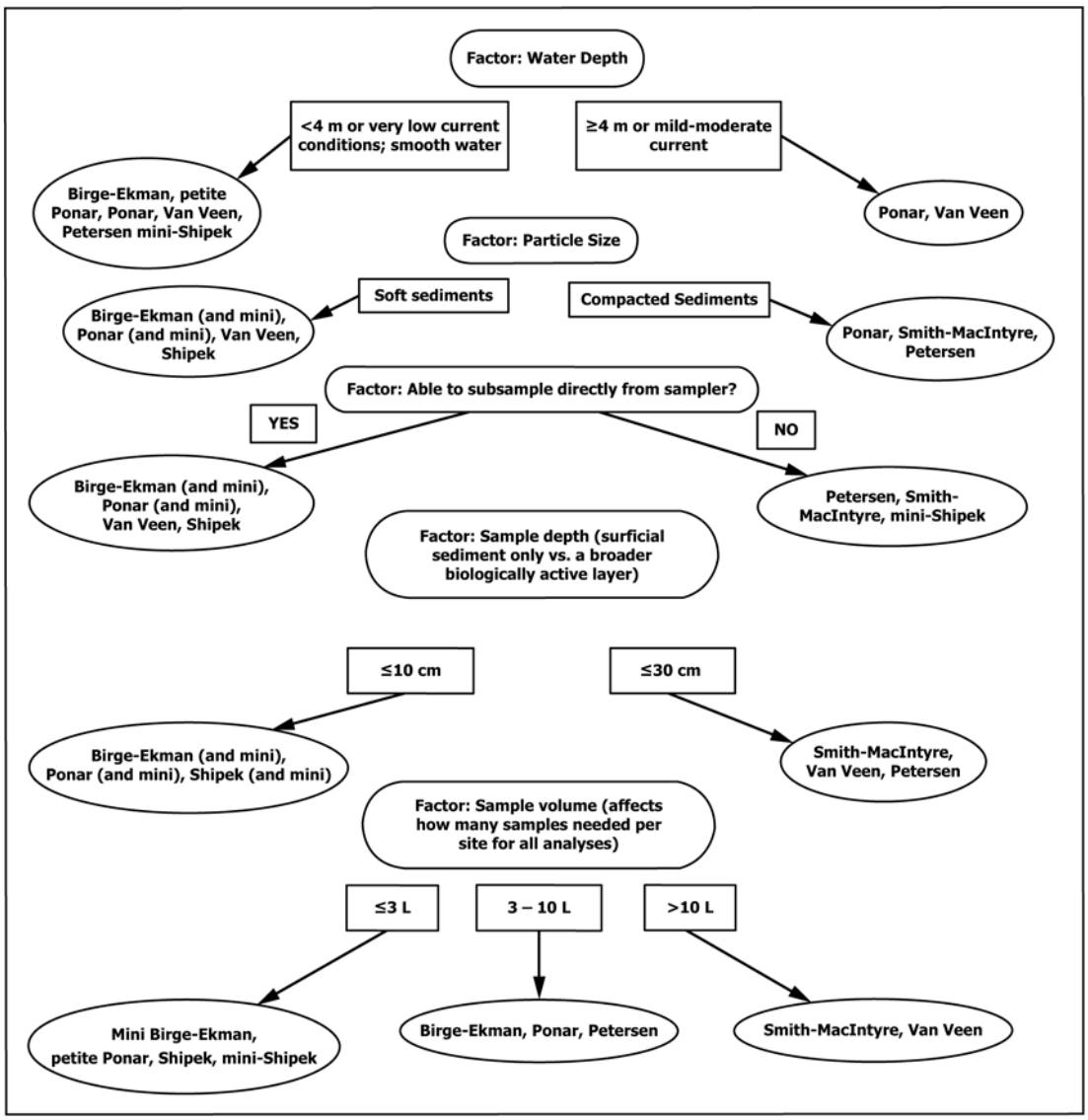

FIG. 4 Flowchart for Selecting Appropriate Grab Samplers Based on Site Specific or Design Factors (USEPA 2001 [1])

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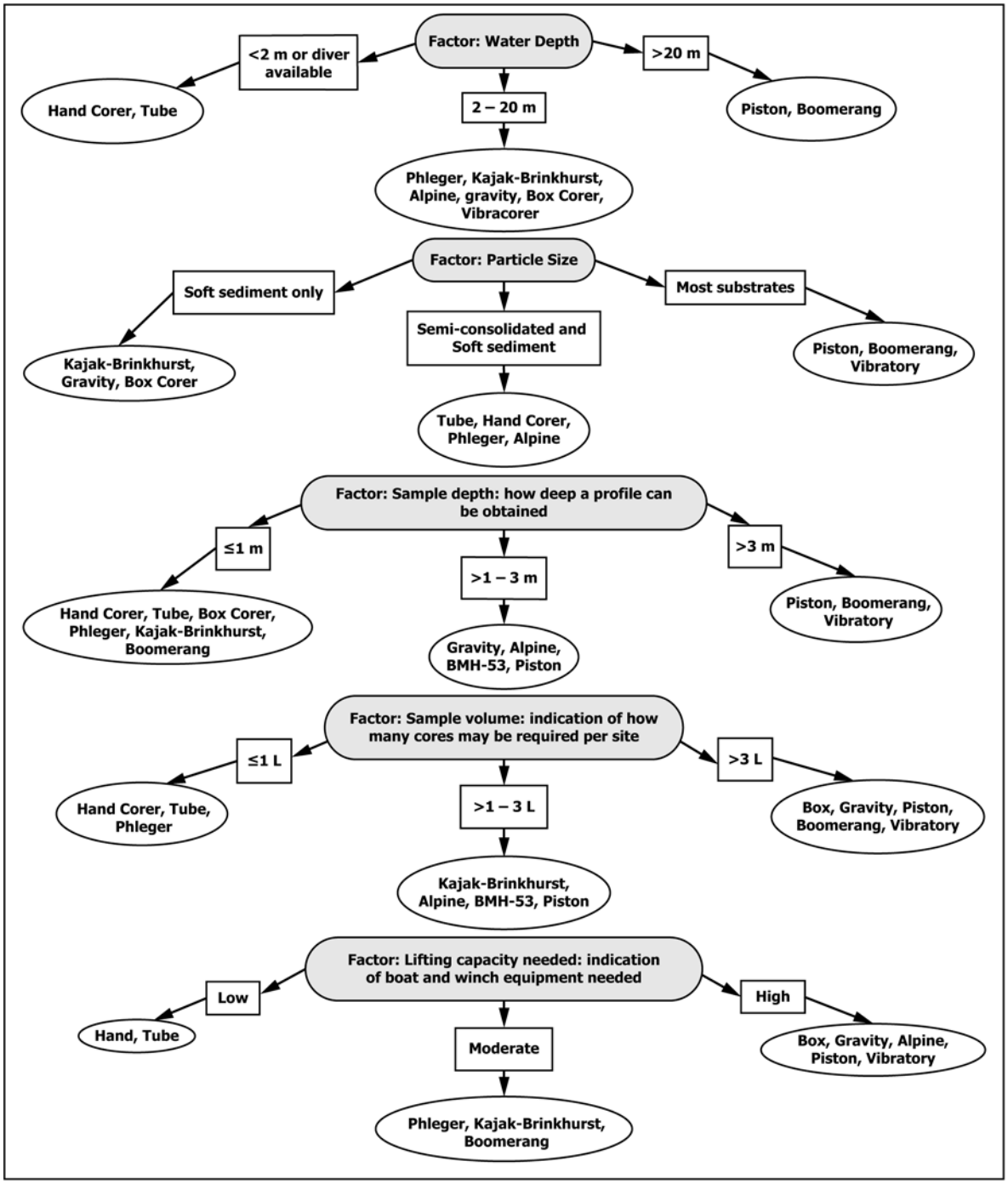

FIG. 5 Flowchart for Selecting Appropriate Core Samplers Based on Site-specific Factors (USEPA 2001 (1))

primarily interested in characterizing recent environmental impacts in lakes, estuaries, and coastal waters, and therefore sample surface sediments (for example, Long et al. 1996 (32)). Other programs (for example, dredged material characterization studies conducted for USEPA and the US Army Corps of Engineers), are concerned with the vertical distribution of contaminants in sediment to be dredged and therefore seek to characterize a sediment column (USEPA/USACE, 1991(33), 1998 (35)). Each of these applications might use different sampling devices.

10.1.5 Related to study objectives, another important factor in selecting a sampler is desired depth of sediment penetration. For monitoring and assessment studies where historical contamination is not the focus, the upper 10 to 15 cm is typically the horizon of interest. For example, Test Method E1706 states sediment should be collected from a depth that will represent expected exposure. Generally, these are the most recently deposited sediments, and most epifaunal and infaunal organisms are found in this horizon. To minimize disturbance of the upper layer during sampling, a minimum penetration depth of

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6 to 8 cm is suggested, with a penetration depth of 10 to 15 cm being preferred. However, if sediment contamination is being related to organism exposures (for example, benthic macroinvertebrates or fish) then more precise sampling of sediment depths might be needed, such as with a core sampler. The life history and feeding habits of the organisms (receptors) of concern should be considered. For example, some organisms (for example, shrimp, rotifers) might be epibenthic and are only exposed to surficial sediments (for example, 0 to 1 cm), while others (for example, amphipods, polychaetes) that are infaunal irrigators might receive their primary exposure from sediments that are several centimeters in depth. Relating contaminant levels that occur in sediment layers where resident organisms are not exposed might produce incorrect conclusions (Lee 1991(**17**)).

10.1.6 Sampling of the surface layer provides information on the horizontal distribution of parameters or properties of interest for the most recently deposited material. Information obtained from analysis of surface sediments can be used, for example, to map the distribution of a chemical contaminant in sediments across a specific body of water (for example, lake, embayment, estuary). A sediment column, including both the surface sediment layer and the sediment underneath this layer, is collected to study historical changes in parameters of interest (as revealed through changes in their vertical distribution), and to characterize sediment quality with depth.

10.1.7 Once study objectives and the general type of sampler have been identified, a specific sampler is selected based on knowledge of the bathymetry and areal distribution of physically different sediment types at the sampling site. Therefore, this information should be gathered during the initial planning stage of the sample collection effort (see 9.7.2).

10.1.8 The quantity of sediment to be collected at each sampling station may also be an important consideration in the selection of a sampling device (see also 9.6.6). The required quantity of sediment typically depends on the number and type of physicochemical and biological tests to be carried out. Table 3 provides a summary of typical sediment volumes needed for different analyses.

10.1.9 Regardless of the type of sampler used, it is important to follow the standard operating procedures specific to each device. Before retrieving the sample, the outside of the sampling device should be carefully rinsed with water from the sampling station. Between each sampling event, the sampling device should be cleaned, inside and out, by dipping the sampler into and out of the water rapidly or by washing with water from the location being sampled. More rigorous between-sample cleaning of the sampler (for example, chemical decontamination or washing with soap) might be required, depending on the nature of the investigation (see 10.5).

10.1.10 To minimize cross-contamination of samples and to reduce the amount of equipment decontamination required, it might be prudent to sample reference stations (that is, relatively clean stations) first, followed by test stations. If certain stations are known to be heavily contaminated, it might be prudent to sample those stations last when sampling many locations at one time.

10.2 *Types of Sediment Samplers:*

10.2.1 There are three main types of sediment sampling devices: grab samplers, core samplers, and dredge samplers. Grab samplers (Annex A1) are typically used to collect surficial sediments for the assessment of the horizontal distribution of sediment characteristics. Core samplers (Annex A1) are typically used to sample thick sediment deposits, or to collect sediment profiles for the determination of the vertical distribution of sediment characteristics or to characterize the entire sediment column. Dredge samplers are used primarily to collect benthos (Annex A1). Dredges cause disruption of sediment and pore water integrity, as well as loss of fine-grained sediments. For these reasons, only grab and core samplers are recommended for sediment physicochemistry or toxicity evaluations. Since many grab samplers are appropriate for collecting benthos as well (Klemm et al. 1990 (**31**) and Guide D4387), grab samplers are likely to be more useful than dredges in sediment quality assessments. Therefore, dredges are not considered further in the following sections.

10.2.2 Advantages and disadvantages of various grab and core samplers are summarized in Tables A1.1-A1.4 in Annex A1 and are discussed briefly in the following sections. Figs. 4 and 5 and Table 7 provide recommendations regarding the type of sampler that would be appropriate given different study objectives. For many study objectives either cores or grab samplers can be used, however, in practice, one will often be

TABLE 7 Recommendations for Selecting Appropriate Sediment Sampling Devises Based on the Study Objectives (USEPA 2001 (1))

Grab or core samplers are preferred over dredges for collecting surficial sediments for physicochemical or toxicity analyses. Dredges might be acceptable for collecting macroinvertebrates.
Grab samplers are recommended for surficial sediment analyses where accurate resolution of surficial sediment depths is not necessary. Core samplers are recommended for: (a) assessments requiring accurate surficial sediment depth resolution, (b) historical sediment analyses, (c) detailed sediment quality studies of vertical sediment profiles, to characterize sediment quality at depth, (d) when characterizing thick sediment deposits (such as shoals to be excavated), and/or (e) where it is important to maintain an oxygen-free environment.
In sand, gravel, firm clay, or till sediments, grab samplers might be preferred over core samplers (when only surface material needs to be collected and samples at depth are not necessary) because the latter are often less efficient in these sediment types.
Ponar, VanVeen, or Ekman samplers are commonly used and generally preferred for grab sampling. Ekman samplers, however, are less efficient in deep waters.
The Kajak-Brinkhurst corer is a common core sampler for soft, fine grained sediments where large volumes or deep cores are not needed. The Phleger corer is commonly used for a variety of sediments including peat and plant roots but is not appropriate where large volumes or deep cores are needed.
Box corers are especially recommended for: (a) studies of the sediment-water interface; (b) collecting larger volumes of sediment from a given depth (generally less than one meter depth, however); (c) for in-situ studies involving interstitial water characterization; and (d) collecting subsamples for different analyses from the same station.
Vibracorers are recommended for studies requiring deep cores (> 1 m), or where sediment consists of very compacted or large grained material (for example, gravel).

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preferred over the other depending on other constraints such as amount of sample required for analyses and equipment availability.

10.2.3 *Grab Samplers:*

10.2.3.1 Grab samplers consist either of a set of jaws that shut when lowered into the surface of the bottom sediment, or a bucket that rotates into the sediment when it reaches the bottom (Annex A1). Grab samplers have the advantages of being relatively easy to handle and operate, readily available, moderately priced, and versatile in terms of the range of substrate types they can effectively sample.

10.2.3.2 Of the grab samplers, the Van Veen, Ponar, and Petersen are the most commonly used. These samplers are effective in most types of surface sediments and in a variety of environments (for example, lakes, rivers, estuaries, and marine waters). In shallow, quiescent water, the Birge-Ekman sampler also provides acceptable samples and allows for relatively nondisruptive sampling. However, this sampler is typically limited to soft sediments. The Van Veen sampler, or the modified Van-Veen (Ted Young), is used in several national and regional estuarine monitoring programs, including the NOAA National Status and Trends Program, the USEPA Environmental Monitoring and Assessment Program (EMAP), and the USEPA National Estuary Program, because it can sample most types of sediment, is less subject to blockage and loss of sample than the Petersen and Ponar samplers, is less susceptible to forming a bow wave during descent, and provides generally high sample integrity (Klemm et al. 1990 (**31**)). The support frame further enhances the versatility of the VanVeen sampler by allowing the addition of either weights (to increase penetration in compact sediments) or pads (to provide added bearing support in extremely soft sediments). However, this sampler is relatively heavy and requires a power winch to operate safely (GLNPO, 1994 (**11**)).

10.2.3.3 As shown in Annex A1, grab sampler capacities range from about 0.5 to 75 L. If a sampler does not have sufficient capacity to meet the study plan requirements, additional samples can be collected and composited to obtain the necessary sample volume. Grab samplers penetrate to different depths depending on their size, weight, and the bottom substrate. Heavy, large volume samplers such as the Smith-McIntyre, large Birge-Ekman, Van Veen, and Petersen devices can effectively sample to a depth of 30 cm. These samplers might actually sample sediments that are too deep for certain study objectives (that is, not reflective of recently deposited sediments). Smaller samplers such as the small Birge-Ekman, standard and petite Ponar, and standard Shipek devices can effectively collect sediments to a maximum depth of 10 cm. The mini-Shipek can sample to a depth of 3 cm.

10.2.3.4 Another consideration in choosing a grab sampler is how well it protects the sample from disturbance and washout. Grab samples are prone to washout which results in the loss of surficial, fine grained sediments that are often important from a biological and contaminant standpoint. The Ponar, Ted-Young modified grab, and Van Veen samplers are equipped with mesh screens and rubber flaps to cover the jaws. This design allows water to pass through the samplers during descent, reducing disturbance from bow waves at the sediment-water interface. The rubber flaps also serve to protect the sediment sample from washout during ascent. However, meshed screens on samplers may result in wash out of sample after collection, and rubber flaps may be difficult to decontaminate between samples.

10.2.3.5 The use of small or lightweight samplers, such as the small Birge-Ekman, petite Ponar, and mini-Shipek, can be advantageous because of easy handling, particularly from a small vessel or using only a hand line. However, these samplers should not be used in strong currents or high waves. This is particularly true for the Birge-Ekman sampler, which requires relatively calm conditions for proper performance. Lightweight samplers generally have the disadvantage of being less stable during sediment penetration. They tend to fall to one side due to inadequate or incomplete penetration, resulting in unacceptable samples.

10.2.3.6 In certain very shallow water applications, such as a stream assessment, it might be difficult to use even a lightweight sampler to collect a sample. In these cases, sediment can be collected from depositional areas using a shovel or other hand implement. However, such sampling procedures are discouraged as a general rule and the use of a hand corer or similar device is preferred (see 10.2.4).

10.2.3.7 Fig. 4 summarizes appropriate grab samplers based on two important site factors, depth and sediment particle size. This figure also indicates appropriate grab samplers depending on certain common study constraints such as sample depth and volume desired, and the ability to subsample directly from the sampler (see 11.4 and Guide D4387). Based on all of these factors, the Ponar or Van Veen samplers are perhaps the most versatile of the grab samplers, hence their common usage in sediment studies.

10.2.3.8 Careful use of grab samplers is required to minimize problems such as loss of fine-grained surface sediments from the bow wave during descent, mixing of sediment layers upon impact, lack of sediment penetration, and loss of sediment from tilting or washout upon ascent (USEPA 2001(**1**); Environment Canada, 1994 (**2**); Baudo, 1990 (**47**); Golterman et al., 1983 (**48**); Plumb,1981 (**49**)). When deploying a grab sampler, the speed of descent should be controlled, with no "free fall" allowed. In deep waters, a winching system should be used to control both the rate of descent and ascent. A ball-bearing swivel should be used to attach the grab sampler to the cable to minimize twisting during descent. After the sample is collected, the sampling device should be lifted slowly off the bottom, then steadily raised to the surface at a speed of about 30 cm/sec (Environment Canada, 1994 (**2**)).

10.2.4 *Core Samplers:*

10.2.4.1 Core samplers (corers) are used: (*1*) to obtain sediment samples for geological characterizations and dating, (*2*) to investigate the historical input of contaminants to aquatic systems and, (*3*) to characterize the depth of contamination at a site. Corers are an essential tool in sediments in which 3-dimensional maps of sediment contamination are necessary. Table A1.2 discusses some of the advantages and disadvantages of common corers.

10.2.4.2 Core devices should be used for projects in which it is important to maintain the integrity of the sediment profile,

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because these devices are considered to be less disruptive than dredge or grab samplers. Core samplers should also be used where it is important to maintain an oxygen-free environment because they limit oxygen exchange with the air more effectively than grab samplers. Cores should also be used where thick sediment deposits are to be representatively sampled (for example, for dredging projects).

10.2.4.3 One limitation of core samplers is that the volume of any given depth horizon within the profile sample is relatively small. Thus, depending on the number and type of analyses needed, repetitive sampling at a site might be required to obtain the desired quantity of material from a given depth. Some core samplers are prone to "plugging" or "rodding" where the friction of the sediment within the core tube prevents it from passing freely and the core sample is compressed or does not sample to the depth required. This limitation is more likely with smaller diameter core tubes and heavy clay sediments. Except for piston corers and vibracorers, there are few core devices that function efficiently in substrates with significant proportions of sand, gravel, clay, or till.

10.2.4.4 Coring devices are available in various designs, lengths, and diameters (Annex A1). With the obvious exception of hand corers, there are only a few corers that can be operated without a mechanical winch. The more common of these include the standard Kajak-Brinkhurst corer, suitable for sampling soft, fine-grained sediments, and the Phleger corer, suitable for a wider variety of sediment types ranging from soft to sandy, semi-compacted material, as well as peat and plant roots in shallow lakes or marshes (Mudroch and Azcue, 1995 (**46**)). The Kajak-Brinkhurst corer uses a larger core tube, and therefore recovers a greater quantity of sediment, than the Phleger corer. Both corers can be used with different liner materials including stainless steel and PVC. Stainless steel liners should not be used if trace metal contamination is an issue.

10.2.4.5 Gravity corers are appropriate for recovering up to 3 m long cores from soft, fine-grained sediments. Recent models include stabilizing fins on the upper part of the corer to promote vertical penetration into the sediment, and weights that can be mounted externally to enhance penetration (Mudroch and Azcue, 1995 (**46**)). A variety of liner materials are available including stainless steel; Lexan®, and PVC. For studies in which metals are a concern, stainless steel liners should not be used.

10.2.4.6 Vibracorers are perhaps the most commonly used coring device in the United States because they collect deep cores in most types of sediments, yielding excellent sample integrity. Vibracorers are one of the only sampling devices that can reliably collect thick sediment samples (up to 10 m or more). Some programs that rely on vibracorers include the Puget Sound Estuary Program, the USEPA Great Lakes National Program ARCS Program (GLNPO 1994 (**11**)), and the Dredged Materials Management Program. Note that the vibratory action of a vibracore can lead to vertical transport of fines along the wall of the core tube resulting in smearing of the sample. Additionally, unconsolidated materials can be mixed (for example, recently place or capped materials). Consequently, vibracoring may not be appropriate in cases where higher resolution sampling is required in "loose" materials.

10.2.4.7 Vibracorers have an electric-powered, mechanical vibrator located at the head end of the corer which applies thousands of vertical and horizontal vibrations per minute to help penetrate the sediment. A core tube and rigid liner (preferably of relatively inert material such as cellulose acetate butyrate) of varying diameter depending on the specific vibrator head used, is inserted into the head and the entire assembly is lowered in the water. Depending on the horsepower of the vibrating head and its weight, a vibracorer can penetrate very compact sediments and collect cores up to 6 m long. For example, the ARCS program in the Great Lakes uses a Rossfelder® Model P-4 Vibracorer (Rossfelder Corporation, La Jolla, CA) to collect cores up to 6 m in length; however, this particular model is relatively heavy. Therefore, use of a heavy vibracorer requires a large vessel to maintain balance and provide adequate lift to break the corer out of the sediment and retrieve it (GLNPO, 1994 (**11**); PSEP, 1997a(**34**)).

10.2.4.8 When deployed properly, box corers can obtain undisturbed sediment samples of excellent quality. The basic box corer consists of a stainless steel box equipped with a frame to add stability and facilitate vertical penetration on low slopes. Box corers should be used in studies of the sediment-water interface or when there is a need to collect larger volumes of sediment from the depth profile. Because of the heavy weight and large size of almost all box corers, they can be operated only from a vessel with a large lifting capacity and sufficient deck space. Sediment inside a box corer can be subsampled by inserting narrow core tubes into the sediment. The tubes should be machine cut so that the opening is square with the tube shaft, and the ends of the tube should be carefully milled to reduce smearing of the sample on the inside surface of the tube and to improve the ease of penetration of the tube. Core tubes are an ideal sampler for obtaining acceptable subsamples for different analyses at a given station. Carlton and Wetzel (1985 (**50**)) describe a box corer that permits the sediment and overlying water to be held intact as a laboratory microcosm under either the original in situ conditions or other laboratory controlled conditions. A box corer was developed that enables horizontal subsampling of the entire sediment volume recovered by the device (Mudroch and Azcue, 1995 (**46**)).

10.2.4.9 Fig. 5 summarizes the core samplers that are appropriate given site factors such as depth and particle size and other study constraints such as sample depth and volume required, and lifting capacity needed to use the sampling device. Given the factors examined for general monitoring studies, the Phleger, Alpine, and Kajak-Brinkhurst corers might be most versatile. For dredged materials evaluations, and projects requiring sediment profile characterizations greater than 3 m in sediment depth, the vibracorer or piston corer are the samplers of choice.

10.2.4.10 Collection of core samples with hand-coring devices should be performed with care to minimize disturbance or compression of sediment during collection. To minimize disruption of the sediment, core samples should be kept as

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stationary and vibration-free as possible during transport. These cautions are particularly applicable to cores collected by divers.

10.2.4.11 The speed of descent of coring devices should be controlled, especially during the initial penetration of the sediment, to minimize disturbance of the surface and to minimize compression due to frictional drag from the sides of the core liner (Guide D4823). In deep waters, winches should be used where necessary to minimize twisting and tilting and to control the rate of both descent and ascent. With the exception of piston corers or vibracorers, which are equipped with their own mechanical impact features, for other corers, only the weight or piston mechanism of the sampler should be used to force it into the sediment. The sampler should be raised to the surface at a steady rate, similar to that described for grab samplers. Where core caps are required, it is essential to quickly and securely cap the core samples when the samples are retrieved. The liner from the core sampler should be carefully removed and kept in a stable position until the samples are processed (see Section 11). If there is little to no overlying water in the tube and the sediments are relatively consolidated, it is not necessary to keep the core sample tubes vertical. If sediment oxidation is a concern (for example, due to potential changes in metal bioavailability or volatile substances in anoxic sediments), then the head space of the core tube should be purged with an inert gas such as nitrogen or argon.

10.3 *Sample Acceptability:*

10.3.1 Only sediments that are correctly collected with grab or core sampling devices should be used for subsequent physicochemical, toxicity, or bioaccumulation testing. Acceptability of grabs can be determined by noting that the samplers were closed when retrieved, are relatively full of sediment (but not over-filled), and do not appear to have lost surficial fines. At shallow stations when multiple composite samples are being taken to retrieve larger sediment volumes, it is not uncommon to drop the dredge into a previous hole. A visual inspection of the sample surface should be done to determine if only surface sediment has been collected. Slight adjustments in location may be necessary if operating with a crane or if using a hand line, moving elsewhere in the boat to operate the sampler. Core samples are acceptable if the core was inserted vertically in the sediment and an adequate depth was sampled.

10.3.2 A sediment sample should be inspected as soon as it is secured. If a collected sample fails to meet any of the conditions listed in the previous paragraph, then the sample might need to be rejected and another sample collected at the station. The location of consecutive attempts should be as close to the original attempt as possible and located in the "upstream" direction of any existing current. Rejected sediment samples should be discarded in a manner that will not affect subsequent samples at that station or other possible sampling stations. Illustrations of acceptable and unacceptable grab samples are provided in Fig. 6.

10.4 *Equipment Decontamination:*

10.4.1 For most sampling applications, site water rinse of equipment in between stations is normally sufficient (PSEP, 1997a(**34**)). However, if one is sampling many stations, including some that could be heavily contaminated, a site water rinse might not be sufficient to minimize cross-contamination of samples among stations. In these cases, it might be necessary to decontaminate all sampling materials in between stations. This would include the sampling device, scoop, spatula, mixing bowls, and any other utensils that come in contact with sediment samples. See 7.2 for additional detail on cleaning equipment. Alternatively, separate sampling equipment could be used at each station.

10.4.2 If sediment can be collected from the interior of the sampling device, and away from potentially contaminated surfaces of the sampler, it might be adequate to rinse with site water between stations. The interior of the sampler needs to be free of any sediment between sampling stations, and should be either rinsed or physically scrubbed. Particular attention should be paid to corners and seams in the sampling device.

10.4.3 If metals or other inorganic compounds are specifically of concern, sampling and handling equipment should be suspended over a tub and rinsed from the top down with 10 % nitric acid using a pump or squirt bottle (USEPA 1993 (**51**), 2001(**1**)). If organic compounds are a specific concern, sampling equipment can be decontaminated using acetone followed by a site water rinse. Wash water from decontamination should be collected and disposed of properly.

10.5 *Field Measurements and Observations:*

10.5.1 Field measurements and observations are important to any sediment collection study, and specific details concerning sample documentation should be included in the study plan.

10.5.2 Measurements and observations should be documented clearly in a bound field logbook (or on pre-printed sample forms). Preferably, a logbook should be dedicated to an individual project. The investigator's name, project name, project number, and book number (if more than one is required) should be entered on the inside of the front cover of the logbook. All entries should be written in indelible ink, and the date and time of entry recorded. Additionally, each page should be initialed and dated by the investigator. At the end of each day's activity, or entry of a particular event if appropriate, the investigator should enter their initials. All aspects of sample collection and handling as well as visual observations and field conditions should be documented in the field logbooks at the time of sample collection. Logbook entries should also include any circumstances that potentially affected sampling procedures or any field preparation of samples. Data entries should be thorough enough to allow station relocation and sample tracking. Because field records are the basis for later written reports, language should be objective, factual, and free of personal opinions or other terminology that might appear inappropriate.

10.5.3 In describing characteristics of samples collected, some cautions should be noted. First, polarized glasses are often worn in the field to reduce glare, however, they can also alter color vision. Therefore, visual examination or characterization of samples should be performed without sunglasses (GLNPO, 1994 (**11**)). Second, descriptions of sediment texture and composition should rely on a texture-by-feel or "ribbon" test in addition to visual determinations (GLNPO, 1994 (**11**)). In this test, a small piece of suspected clay is rolled between

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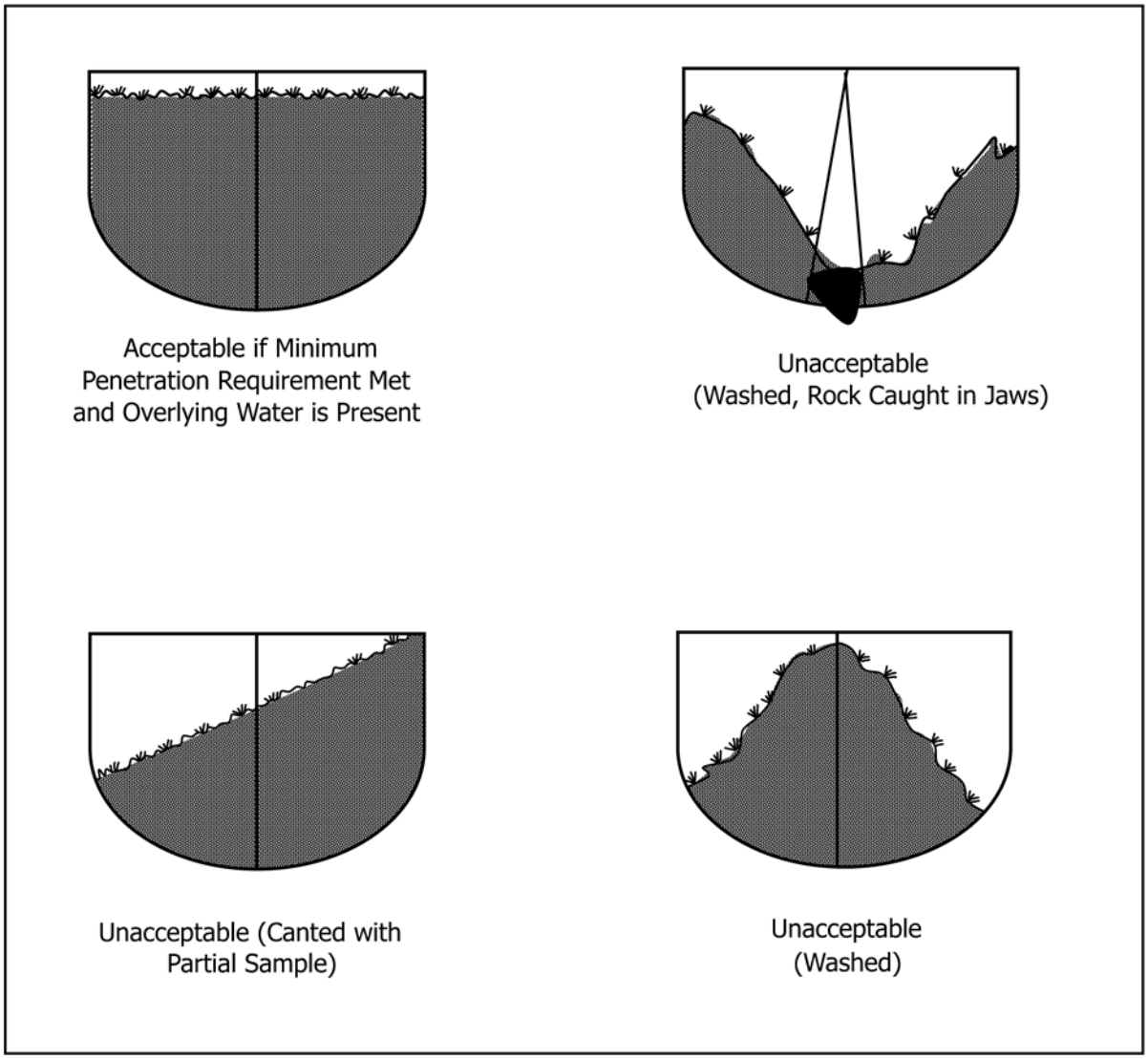

FIG. 6 Illustrations of Acceptable and Unacceptable Grab Samples (USEPA 2001 (1))

the fingers while wearing protective gloves. If the piece easily rolls into a ribbon it is clay; if it breaks apart, it is silt (GLNPO, 1994 (**11**)).

10.5.4 *Documentation of Sample Collection*—Documentation of collection and analysis of sediment and pore-water samples requires all the information necessary to: (*1*) trace a sample from the field to the final result of analysis; (*2*) describe the sampling and analytical methodology; and (*3*) describe the QA/QC program (Mudroch and Azcue 1995(**46**); Keith, 1993 (**52**); Table 8). Poor or incomplete documentation of sample collection can compromise the integrity of the sample(s) and thus, the study. In addition, stations that could not, or were not, sampled should be documented with an explanation. Samples should be accompanied by chain-of-custody forms that identify each sample collected and the analyses to be conducted on that sample. Specific guidance on quality assurance procedures regarding sample chain-of-custody is summarized in Section 15.

11. Field Sample Processing, Transport, and Storage of Sediments:

11.1 The way in which sediment samples are processed, transported, and stored might alter contaminant bioavailability and concentration by introducing contaminants to the sample or by changing the physical, chemical, or biological characteristics of the sample. Manipulation processes often change availability of organic compounds because of disruption of the equilibrium with organic carbon in the pore water and sediment system. Similarly, oxidation of anaerobic sediments increases the availability of certain metals (Di Toro et al. 1990 (**53**); Ankley et al. 1996 (**54**)). Materials and techniques should be selected to minimize sources of contamination and variation,

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TABLE 8 Recommendations on Information to be Documented for Each Sample Collected (PSEP 1997a(34), USEPA 2001 (1))

NOTE 1—Some geological characterization methods might include an odor evaluation of the sediment as this can provide useful information on physicochemical conditions. However, sediment odor evaluation is potentially dangerous depending on the chemicals present in the sediment (Test Method E1706) and should therefore be done cautiously, if at all.

Project title, time and date of collection, sample number, replicate number, site identification (for example, name); station number and location (for example, positioning information);
Water depth and the sampling penetration depth;
Details pertaining to unusual events which might have occurred during the operation of the sampler (for example, possible sample contamination, equipment failure, unusual appearance of sediment integrity, control of vertical descent of the sampler, etc.), preservation and storage method, analysis or test to be preformed;
Estimate of quantity of sediment recovered by a grab sampler, or length and appearance of recovered cores;
Description of the sediment including texture and consistency, color, presence of biota or debris, presence of oily sheen, changes in sediment characteristics with depth, and presence/location/thickness of the redox potential discontinuity (RPD) layer (a visual indication of black is often adequate for documenting anoxia);
Photograph of the sample is desirable, especially longitudinally-sectioned cores, to document stratification;
Deviations from approved work plans or SOPs.

and sample treatment before testing should be as consistent as possible. A flowchart is presented in Fig. 7 that summarizes common sediment processing procedures discussed in this section as well as issues and objectives relevant to each processing step.

11.2 *Sample Containers:*

11.2.1 Any material that is in contact with a field sample has the potential to contaminate the sample or adsorb components from the sample. For example, samples can be contaminated by zinc from glassware, metals from metallic containers, and organic compounds from rubber or plastic materials. The use of appropriate materials, along with appropriate cleaning procedures, can minimize or mitigate interferences from sample containers.

11.2.2 *Container Material:*

11.2.2.1 Equipment and supplies that contact sediments or overlying water should not contain substances that can be leached or dissolved in amounts that adversely affect the test organisms or interfere with chemical or physical analyses. In addition, equipment and supplies that contact sediment or water should be chosen to minimize sorption of test materials from water. Glass, Type 316 stainless steel, nylon, high-density polyethylene, polypropylene, polycarbonate, and fluorocarbon plastics should be used whenever possible to minimize leaching, dissolution, and sorption (Test Method E1706). Direct contact between sediment samples and the following substances should be avoided: PVC, natural or neoprene rubber, nylon, talcum powder, polystyrene, galvanized metal, brass, copper, lead, other metal materials, soda glass, paper tissues, and painted surfaces. Table 9Table 10 summarizes the appropriate types of sampling containers and allowable holding times for various types of contaminants associated with sediments.

11.2.2.2 In general, sediments and pore waters with multiple or unknown chemical types should be stored in containers made from high density polyethylene plastic or polytetrafluoroethylene (PTFE) as these materials are least likely to add chemical artifacts or interferences and they are much less fragile than glass. Samples for organic contaminant analysis should be stored in brown borosilicate glass containers with PTFE lid liners. If volatile compounds will be analyzed, containers should have a septum to minimize escape of volatile gases during storage and analysis. Extra containers should be provided for these analyses in the event that re-analysis of the sample is required. If samples are contaminated with photoreactive compounds such as PAHs, exposure to light should be minimized by using brown glass containers or clear containers wrapped tightly with an opaque material (for example, clean aluminum foil). Plastic or acid-rinsed glass containers should be used when the chemicals of concern are heavy metals.

11.2.2.3 In general, anything coming in contact with the sediment during sample collection, processing and subsequent testing should be made of non-contaminating materials. However, in certain cases (for example, in situ testing) it may be necessary to use materials (PVC, fiberglass, etc.) that have a potential to leach contaminants. In such instances it is advisable that such materials be soaked or aged for an extended period of time (for example, 7 days) before use to reduce the amount of contaminants potentially leached from these materials (see 11.2.3.2).

11.2.3 *Container Preparation:*

11.2.3.1 Many vendors have commercially available pre-cleaned containers for a variety of applications. For chemical and toxicological analyses, certified pre-cleaned containers are often a cost-effective way to limit the potential for container contamination of samples. Thus, manufacturer-supplied pre-cleaned containers are often a prerequisite in QAPPs.

11.2.3.2 If new containers are used, materials should be soaked or aged before use (see 7.2, 12.2.2.3, and Test Method E1706).

11.2.3.3 If a sample is to be refrigerated, the container should be filled to the brim to reduce oxygen exposure. This is particularly important for volatile compounds (for example, AVS). If a sample is to be frozen, the container should be filled to no more than about 90 % of its volume (about 10 % headspace) to allow for expansion of the sample during freezing. See 11.5 for preservation and storage conditions for various types of analyses. For studies in which it is important to maintain the collected sediment under anoxic conditions (for example, where metal contamination is of concern), the container should be purged with an inert gas (for example, nitrogen) before filling and then again before capping tightly. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E1706 and Guide E1688).

11.2.3.4 All sediment containers should be properly labeled with a waterproof marker before sampling. Containers should be labeled on their sides in addition to or instead of labeling the

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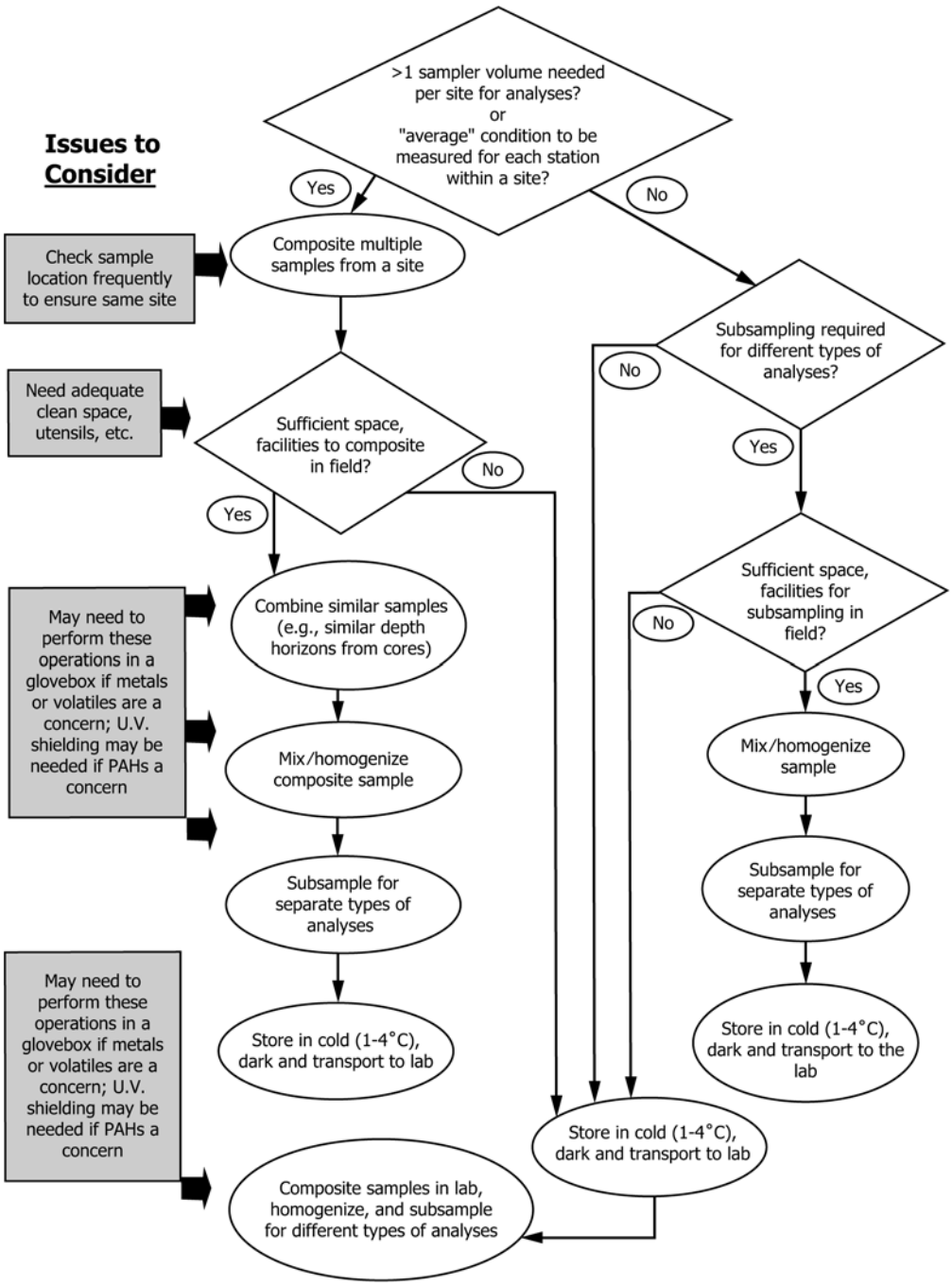

FIG. 7 Flowchart of Suggested Sediment Processing Procedures (USEPA 2001 (1))

lids. Each label should include, at a minimum, the study title, station location or sample identification, date and time of collection, sample type, and name of collector. Blind sample labeling (that is, a sample code) should be used, along with a sample log that identifies information about each sample (see 9.9) to minimize potential analytical bias. Additional information such as required analyses and any preservative used might also be included on the label although this information is typically recorded on the chain-of-custody form (see 9.9 and 15.6). Labeled containers should be stabilized in an upright position in the transport or storage container (see 11.5, Transport and Storage for further information). Extra containers should be carried on each sampling trip.

11.3 *Subsampling and Compositing Samples:*

11.3.1 The decision to subsample or composite sediment samples within or among stations depends on the purpose and objectives of the study, the nature and heterogeneity of the sediments, the volume of sediment required for analytical or toxicity assessment, and the degree of statistical resolution that is acceptable. Subsampling and compositing might be accomplished in the field, if facilities, space and equipment are

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TABLE 9 Recommended Sampling Containers, Holding Times, and Storage Conditions for Common Types of Sediment Analyses (USEPA, 1983 (55);1993(51); 2001 (1))

NOTE 1—P = Plastic; G = Glass; PTFE = Polytetrafluoroethylene; R = refrigerate; F = freeze

Contaminant	Container	Holding Time	Storage Condition
Ammonia	P,G	28 days	R; F
Sulfate	P,G	28 days	R; F
Sulfide	P,G	28 days	R or NaOH; pH>9
Oil and Grease	G	28 days	HCl, pH<2
Mercury	P,G	6 weeks	H_2SO_4, pH<2; R
Metals (except Cr or Hg)	P,G	6 months	HNO_3, pH<2; F
Extractable organics (including phthalates, airosamines, organochlorine pesticides, PCBs, aromatics, isophorone, PAHs, haloethers, chlorinated hydrocarbons, and TCDD)	G, PTFE-lined cap	7 days (until extraction) 30 days (after extraction)	R; F
Purgables (halocarbons and aromatics)	G, PTFE-lined septum	14 days	R; F
Pesticides	G, PTFE-lined cap	7 days (until extraction) 30 days (after extraction)	R; F
Sediment Toxicity (acute and chronic)	P, PTFE	2 weeks[A]	R, dark
Bioaccumulation testing	P, PTFE	2 weeks[A]	R, dark

[A] Holding time might be longer depending on the magnitude an type of contaminants present. Test Methods E1706, E1367 and Guide E1688.

TABLE 10 Recommendations for Subsampling or Compositing Sediment Samples (USEPA 2001 (1))

Overlying water should be siphoned off, not decanted, from grab samplers prior to subsampling.
All utensils that are used to process samples should be made of inert materials such as polytetrafluoroethylene (PTFE) high quality stainless steel, or HDPE.
Subsamples should be collected away from the sides of the sampler to avoid potential contamination.
Sediment samples should be processed prior to long-term storage, within 72 h (and preferably within 24 h) of collection.
Sufficient sample homogenization, prior to placing in containers, is critical for accurate measurements and correct sediment quality determinations.
If rigorous evaluation of metal contamination is a focus of the study, or if anaerobic conditions need to be maintained for other reasons, it might be necessary to homogenize, subsample, and composite samples in an oxygen-free glovebox or other suitable apparatus.
Similar depth horizons or geologic strata should be subsampled when compositing core samples.

available, or alternatively, in a laboratory setting following sample transport Table 10.

11.3.2 *General Procedures:*

11.3.2.1 Subsampling is useful for collecting sediment from a specific depth of a core sample, for splitting samples among multiple laboratories, for obtaining replicates within a sample, or for forming a composite sample.

11.3.2.2 Compositing refers to combining aliquots from two or more samples and analyzing the resulting pooled sample (Keith, 1993 (**52**)). Compositing is often necessary when a relatively large amount of sediment is needed from each sampling site (for instance, to conduct several different physical, chemical or biological analyses). Compositing might be a practical, cost-effective way to obtain average sediment characteristics for a particular site Table 10, but not to dilute a contaminated sample. Also, if an objective of the study is to define or model physicochemical characteristics of the sediment, it might be important not to composite samples because of model input requirements (EPRI, 1999 (**56**)).

11.3.3 *Grab Samples:*

11.3.3.1 If a sediment grab sample is to be subsampled in the laboratory, the sample should be released carefully and directly into a labeled container that is the same shape as the sampler and made of a chemically-inert material (see 11.2 for recommendations on containers). The container needs to be large enough to accommodate the sediment sample and should be tightly sealed with the air excluded.

11.3.3.2 If the grab sample is to be subsampled in the field, it is desirable to subsample from the sampler directly to minimize sediment handling and associated artifacts. Therefore, the sampler should allow access to the surface of the sample without loss of water or fine-grained sediment (see 10.1 for sampler descriptions). This typically dictates the use of a grab sampler with bucket covers that are either removable or hinged to allow access to the surface of the sediment sample (for example, Ponar, VanVeen).

11.3.3.3 Before subsampling from the grab sampler, the overlying water should be removed by slow siphoning using a clean tube near one side of the sampler (WDE, 1995 (**29**); PSEP, 1997a (**34**)). If the overlying water in a sediment sampler is turbid, it should be allowed to settle if possible.

11.3.3.4 The general subsampling and compositing process for grab samples is illustrated in Fig. 8. Subsampling can be performed using a spoon or scoop made of inert, non-contaminating material. Sediment that is in direct contact with the sides of the grab sampler should be excluded as a general precaution against potential contamination from the device. Subsamples may be combined or placed into separate clean, pre-labeled containers. If the sample is to be frozen, it is advisable to leave at least about 10 % head space in the container to accommodate expansion and avoid breakage. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E1706 and Guide E1688).

11.3.3.5 There are two alternatives for compositing sediment samples from grab samplers Fig. 8: (*1*) compositing and homogenizing (mixing) in the field and (*2*) compositing in the field and homogenizing in the laboratory.

11.3.3.6 In some studies (for example, where metals are the contaminants of concern), it might be necessary to subsample a grab sample under oxygen-free conditions to minimize oxidative changes. In these cases, a hand-coring device should be used for subsampling. The core should be inserted immediately upon retrieval of the sampler, then removed and placed into a glove box or bag which is flushed with a constant,

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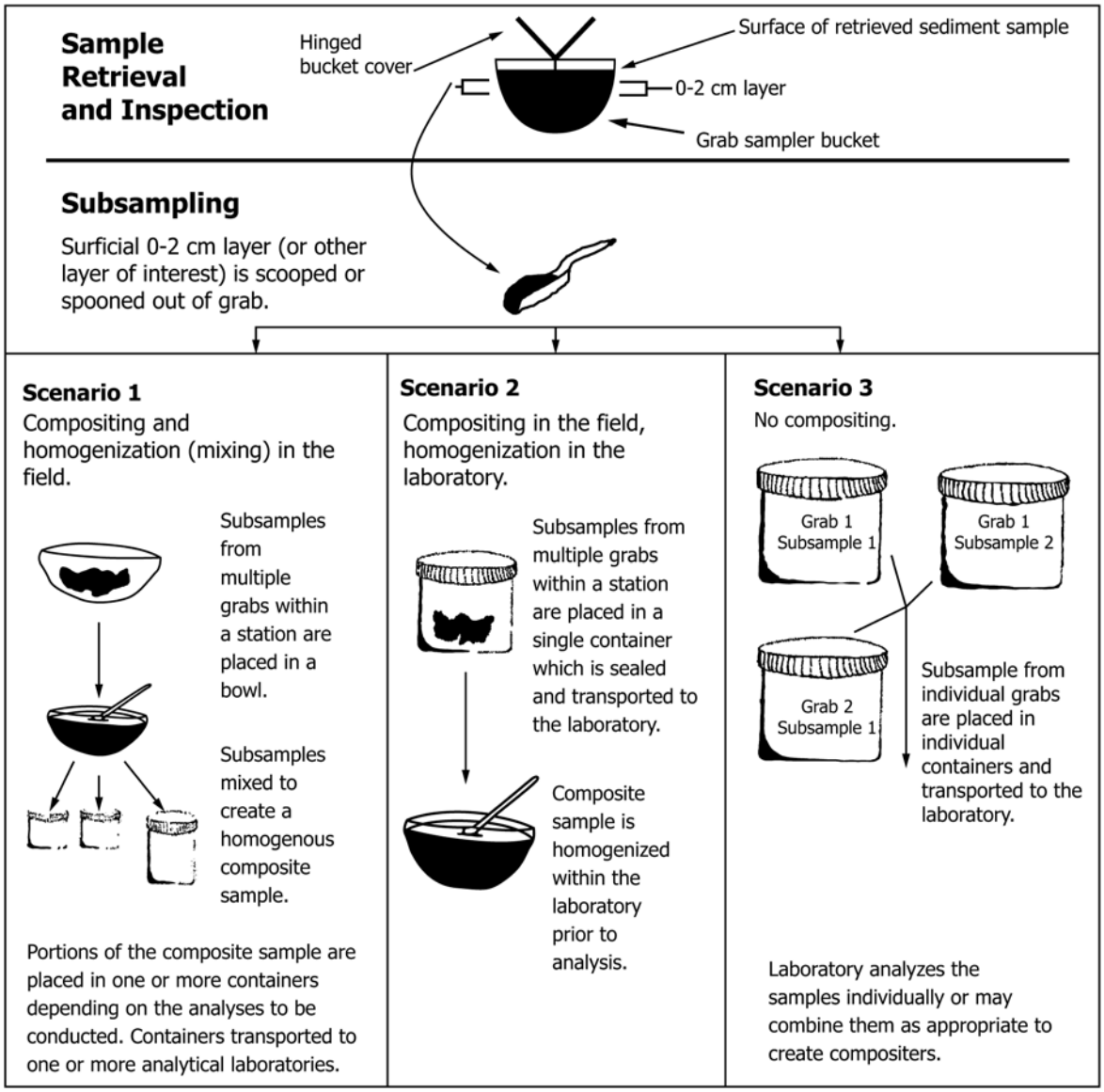

FIG. 8 Alternatives for Subsampling and Compositing Sediment Grab Samples (USEPA 2001(1))

controlled volume of inert gas. The sediment within the core can then be extruded under oxygen-free conditions into deaerated containers. The presence of oxygen during handling and storage might be relatively unimportant (Brumbaugh et al. 1994 (27)) or very important (Besser et al. 1995 (57)), depending on the sediment characteristics, the contaminants of concern, and the study objectives.

11.3.4 *Core Samples:*

11.3.4.1 Subsampling sediment core samples is usually done to focus the assessment on a particular sediment horizon or horizons, or to evaluate historical changes or vertical extent in contamination or sedimentation rates. Whenever subsampling of retrieved sediment cores is required, particularly for analysis of contaminants, the sediment should be extruded from the core liners and subsampled as soon as possible after collection. This can be accomplished in the field if appropriate facilities and equipment are available, or in the laboratory after transport.

11.3.4.2 Systematic subsampling Fig. 9 involves removing the sediment from the core in sections of uniform thickness. Each incremental core section corresponds to a particular sediment depth interval. In remedial dredging and geological applications, longer sections (for example, 25 to 50 cm) are typically used to characterize a site.

11.3.4.3 The depth horizon(s) sampled will depend on the study objectives as well as the nature of the substrate. For toxicological studies, the biologically active layer and sedimentation rates at the site are important factors determining which core sections are sampled. In these studies, subsampling depth intervals may include the 0 to 2 cm layer for recent deposition or greater than the 2-cm layer if the deposition rate is known to be higher, and the 0 to 5 cm or 0 to 15 cm layers for biological activity, depending on resident organisms. Many investigations have project-specific depths corresponding to study requirements, such as dredging depths for navigation or

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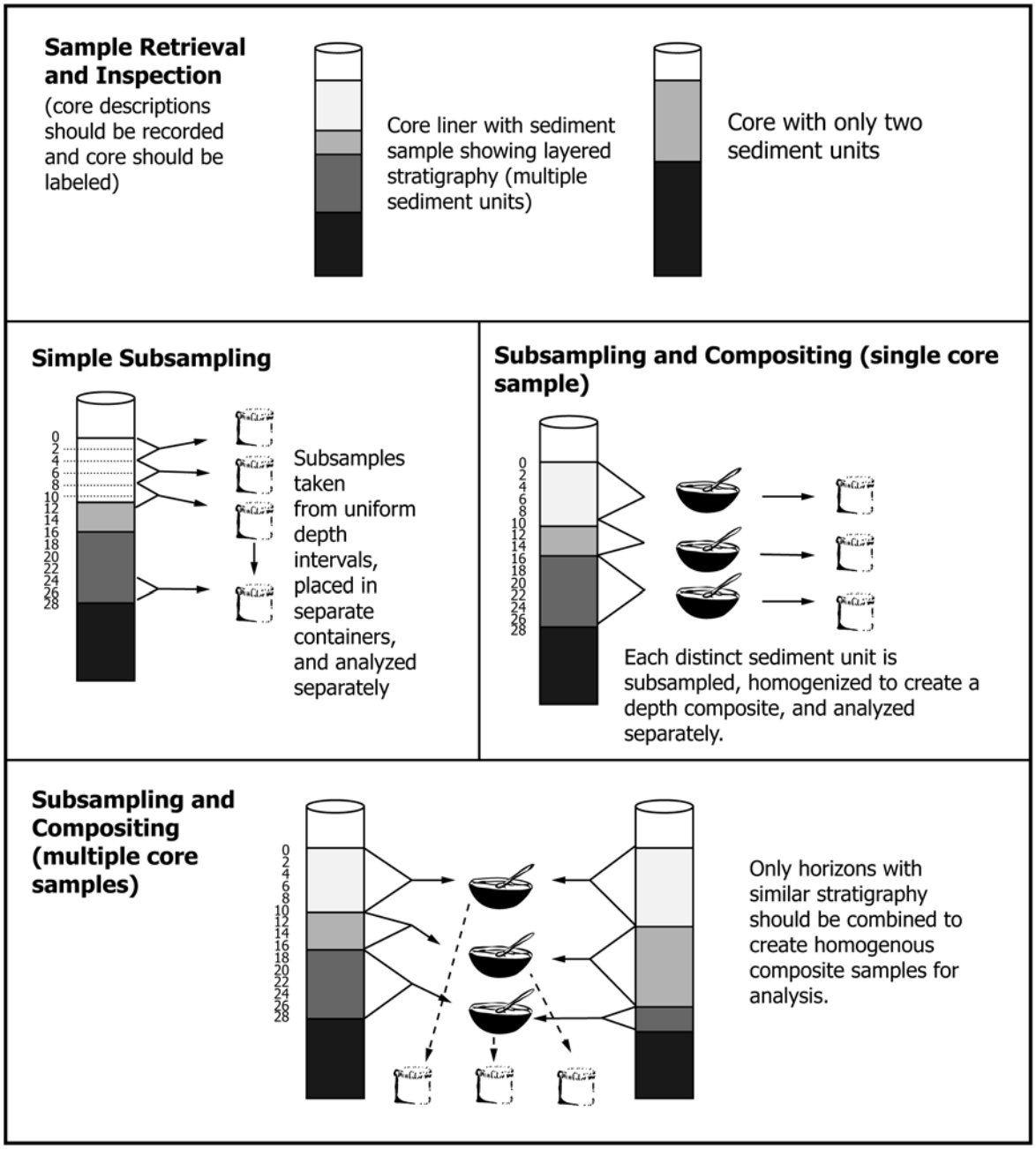

FIG. 9 Alternatives for Subsampling and Compositing Sediment Core Samples (USEPA 2001 (1))

remediation dredging. In many regional or national environmental monitoring programs (for example, USEPA EMAP, NOAA Status and Trends), the uppermost surficial layer is sampled because information on the horizontal distribution of sediment contaminants is desired (USEPA, 2000d (30), Wolfe et al. 1993 (22)).

11.3.4.4 There are various methods for subsampling sediment cores including gradual extrusion, dissection of a core using a jig saw, reciprocating saws, use of a segmented gravity corer, a hand corer, or scoops and spoons. Cutting devices range from stainless steel knives to polytetrafluoroethylene (PTFE) or nylon string. Note that metal saws frequently generate debris that can contaminate a sample. An electric sheet metal cutter has been used on plastic core liners or aluminum core tubes creating a ribbon of material as opposed to chips left behind with a metal saw (David Moore, MEC Analytical, Carlsbad, CA, personal communication).

11.3.4.5 A piston-type extruder that applies upward pressure on the sediment is an instrument commonly used to gradually expose a core for sectioning in some monitoring programs where specific sediment depths have been defined a priori (Kemp et al. 1971 (58)). The capped core liner containing the sediment and overlying water is uncapped at the lower end and placed vertically on top of the piston. The top cap is removed and the water is siphoned off to minimize disturbance of the sediment-water interface. The core liner is then pushed slowly

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down until the surface of the sediment is at the upper end of the liner. Sediment sections are collected by pushing the liner down and cutting the exposed sediment into sections of the desired thickness using a stainless steel or polytetrafluoroethylene (PTFE) cutter (Environment Canada, 1994 (**2**); Mudroch and Azcue, 1995 (**46**)). A 1- to 2-mm outer layer of sediment that has been in contact with the plastic or metal liner should be removed and discarded, if possible, to avoid contamination. Each sediment subsample should be placed into a labeled, clean, and chemically-inert container, or, if subsamples are being composited, into an appropriately sized mixing bowl. The size of the container should be as close to the volume of the sediment as possible to minimize the head space in the container. If it is desirable to maintain an oxygen-free environment during subsampling, then all handling or manipulations should take place in a glove box or bag filled with an inert gas and modified to accommodate the core liner through an opening (Environment Canada, 1994 (**2**); Mudroch and MacKnight, 1994 (**36**)).

11.3.4.6 Cores of more consolidated material can be mounted onto a horizontal U-shaped rail and the liner cut using a saw mounted on a depth-controlling jig. The final cut can then be made with a sharp knife to minimize contamination of the sediment by liner material, and the core itself can be sliced with polytetrafluoroethylene (PTFE) or nylon string. The core then becomes two D-shaped halves that can be easily inspected and subsampled (**46**). Sediment in contact with the saw blade should not be used for toxicity tests or metals analyses due to potential contamination from the saw blade. Another alternative for sectioning and subsampling is a segmented gravity corer described by Aanderaa Instruments of Victoria, BC, Canada. The core tube of the sampler consists of a series of rings placed on top of one another. Subsampling is carried out by rotating the rings around its other axis so that it cuts sediment layers of similar thickness. This segmented core tube is suitable for sampling fine-grained sediments and allows one person in the field to subsample the core into 1-cm sections (Mudroch and Azcue 1995 (**46**)).

11.3.4.7 Sediment from box-core samples can be effectively subsampled with a small hand corer after the overlying water has been carefully siphoned off and discarded. Hand corers with small inner diameters less than 3 cm tend to compact sediments, so this equipment needs to be used with care. Spoons or scoops have also been used to subsample surface sediments from a box corer (Environment Canada, 1994 (**2**)).

11.3.4.8 Like grab samples, core samples may be composited or subsampled in the field or laboratory after evaluating them for acceptability. Although there might be occasions when it is desirable to composite incremental core depths, only horizons of similar stratigraphy should be composited. Depending on the study objectives and desired sampling resolution, individual horizons within a single core can be homogenized to create one or more "depth composites" for that core, or corresponding horizons from two or more cores might be composited Fig. 9. Composite samples should be homogenized before analysis or testing.

11.4 *Homogenization:*

11.4.1 Homogenization refers to the complete mixing of sediment to obtain consistency of physicochemical properties throughout the sample before using in analyses. Homogenization is typically performed on individual samples, as well as on composited samples and can be done either in the field or the laboratory Table 11.

11.4.2 Depending on the objective of the study, unrepresentative materials (for example, twigs, shells, leaves, stones, wood chips and sea grass) might be removed and documented before homogenization (see 12.3 for techniques to remove unrepresentative material). The need for removal of larger matter depends on the analyses to be conducted.

11.4.3 Mixing should be performed as quickly and efficiently as possible, because prolonged mixing can alter the particle-size distribution in a sample and cause oxidation of the sediments (Ditsworth et al. 1990 (**60**); Stemmer et al. 1990a,b (**61**), (**62**)). This can alter the bioavailability of contaminants, particularly metals, by increasing or decreasing their availability Ankley et al. 1996 (**54**)). If metal contaminants or volatile chemicals are a concern, samples should be mixed in a glove box under an inert atmosphere and quickly partitioned into sample containers for analysis.

11.4.4 Homogenate replicates consist of two or more subsamples, taken from different locations within a mixed sample, and then comparing analytical results of the replicate samples (sometimes called a split sample). After the sediment has been homogenized, it is generally partitioned among sample containers. Partitioning sediments for chemical or biological testing may be accomplished using various methods. In one method, a number of small portions are removed from random locations in the mixing container and distributed randomly in all sample jars until the appropriate volume of sediment is contained in each sample jar for each analysis. During distribution, the sediment can be periodically mixed using a glass rod or porcelain spatula to minimize stratification effects due to differential settling, especially if the sediment is prone to rapid settling. An alternative is to use a splitter box designed to contain and then divide the homogenized sediment.

11.5 *Sample Transport and Storage:*

11.5.1 Transport and storage methods should be designed to maintain structural and chemical qualities of sediment samples. Sediments collected using grab samplers are usually transferred from the sampler to containers that may or may not serve

TABLE 11 Recommendations for Homogenizing Sediment Samples (USEPA 2001 (1))

Use a sufficiently large, precleaned glass or stainless steel mixing bowl to homogenize the sample.
Use clean glass polyethylene, or stainless steel implements (for example, spoon) to mix sediment.
Mixing should be performed as quickly and efficiently as possible while attempting to reduce oxidation of the sample.
Intensive manual mixing of wet sediment, in a suitably large container, is usually sufficient to homogenize the sample Ingersoll and Nelson, 1990 (**59**) .
Regardless of the mixing method selected, the effectiveness of the method should be demonstrated using a homogenate replicate

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as the storage container. The containers might be stored temporarily in the field or they might be transported immediately to a laboratory for storage. If sediment core samples are not sectioned or subsampled in the field, they may be stored upright, in the core liner, for intact transportation to the laboratory. If sectioning or subsampling takes place in the field, then the subsamples may also be transferred to sample containers and stored temporarily. The sample containers with the field-collected sediments are then placed into a transport container and shipped to the laboratory. Proper storage conditions Table 9 should be achieved as quickly as possible after sampling. For those parameters that are preserved via refrigeration (for example, toxicity or bioaccumulation tests), samples should be stored in the field in refrigerated units on board the sampling vessel or in insulated containers containing ice or frozen ice packs. Sediment samples should never be frozen for toxicity or bioaccumulation testing (Test Method E1706 and Guide E1688).

11.5.2 For samples that can be preserved via freezing (for example, some metal and organic chemical analyses), dry ice can be used to freeze samples for temporary storage and transport (USEPA, 1983 (**55**), 1993 (**51**)). Pelletized dry ice has been used effectively to store core samples. It is important to know chilling capacities and efficiencies to determine that temperature regulation is adequate. Care should be taken to prevent refrigerated samples from freezing and to keep frozen samples from thawing. Freezing changes the sediment volume depending on the water content, and it permanently changes the structure of the sediment and potentially alters the bioavailability of sediment associated contaminants (Test Method E1706).

11.5.3 Logistics for sample transport will be specifically tailored to each study. In some cases it is most efficient to transfer samples to a local storage facility where they can be either frozen or refrigerated. Depending on the logistics of the operation, field personnel can transport samples to the laboratory themselves or can use an overnight courier service. If a freight carrier is employed, the user needs to be aware of any potentially limiting regulations (for example, regarding the use of ice or dry ice). Samples should be cooled to that temperature before placement in the transport container. Light should be excluded from the transport container.

11.5.4 Core samples should be transported as intact core liners (tubes). Before sample transport, the entire space over the sediment in the core liner should be filled with site water, and both ends of the core liner should be completely sealed to prevent mixing of the sediment inside. The cores should be maintained in an upright position particularly if the sample is not highly consolidated material, and secured in either a transport container (for example, cooler or insulated box) with ice or ice packs, or in a refrigerated unit that can maintain a temperature near 4°C (Environment Canada, 1994 (**2**)). If the transport container cannot accommodate long core samples such as from vibracorers or piston corers (core liners > 1 m), then the core samples can be cut into 1-m lengths, and the ends securely capped such that no air is trapped inside the liners (see 11.4).

11.5.5 Impregnating unconsolidated sediment cores with epoxy or polyester resins will preserve sediment structure and texture (Ginsburg et al. 1966 (**63**); Crevello et al. 1981 (**64**)), but not the chemical characteristics of the sediment. Therefore, this procedure should not be used for transporting or storing sediment samples for chemical characterization or biological testing (Environment Canada, 1994 (**2**)).

11.6 *Sample Holding Times:*

11.6.1 Because the chemicals of concern influencing sediment characteristics are not always known, it is desirable to hold the sediments after collection in the dark at 4°C (Test Method E1706). Traditional convention has held that toxicity or bioaccumulation tests should be started as soon as possible following collection from the field, although actual recommended storage times range from two weeks (USEPA 2001 (**1**)) to less than eight weeks (USEPA-USACE 1998 (**65**)). Discrepancies in recommended storage times reflected a lack of data concerning the effects of long-term storage on the physical, chemical, and toxicological characteristics of the sediment. However, numerous studies have recently been conducted to address issues related to sediment storage (Dillon et al., 1994 (**66**); Becker et al., 1995 (**67**), Carr and Chapman, 1995 (**68**), Moore et al., 1996 (**69**), Sarda and Burton, 1995(**70**), Sijm et al., 1997 (**71**), DeFoe and Ankley, 1998 (**72**)). The conclusions and recommendations offered by these studies vary substantially and appear to depend primarily upon the type or class of chemical(s) present. Considered collectively, these studies suggest that the recommended guidance that sediments be tested sometime between the time of collection and 8 weeks storage is appropriate. Additional guidance is provided below.

11.6.2 Extended storage of sediments that contain high concentrations of labile chemicals (for example, ammonia, volatile organics) may lead to a loss of these chemicals and a corresponding reduction in toxicity. Under these circumstances, the sediment should be tested as soon as possible after collection, but not later than within two weeks (Sarda and Burton, 1995 (**70**)). Sediments that exhibit low-level to moderate toxicity can exhibit considerable temporal variability in toxicity, although the direction of change is often unpredictable (Carr and Chapman, 1995 (**68**); Moore et al., 1996 (**69**); DeFoe and Ankley, 1998 (**72**). For these types of sediments, the recommended storage time of <8 weeks may be most appropriate. In some situations, a minimum storage period for low-to-moderately contaminated sediments may help reduce variability. For example, DeFoe and Ankley, 1998 (**72**) observed high variability in survival during early testing periods (for example, <2 weeks) in sediments with low toxicity. De Foe and Ankley, 1998 (**72**) hypothesized that this variability partially reflected the presence of indigenous predators that remained alive during this relatively short storage period. Thus, if predatory species are known to exist, and the sediment does not contain labile contaminants, it may be desirable to store the sediment for a short period before testing (for example, 2 weeks) to reduce potential for interferences from indigenous organisms. Sediments that contain comparatively stable compounds (for example, high molecular weight compounds such as PCBs) or which exhibit a moderate-to-high

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level of toxicity, typically do not vary appreciably in toxicity in relation to storage duration (Moore et al., 1996 (**69**), DeFoe and Ankley, 1998 (**72**)). For these sediments, long-term storage (for example, >8 weeks) can be undertaken.

11.6.3 Researchers may wish to conduct additional characterizations of sediment to evaluate possible effects of storage. Concentrations of chemicals of concern could be measured periodically in pore water during the storage period and at the start of the sediment test Kemble et al., 1994(**73**). Ingersoll et al., 1993 (**74**) recommend conducting a toxicity test with pore water within two weeks from sediment collection and at the start of the sediment test. Freezing might further change sediment properties such as grain size or chemical partitioning and should be avoided (Schuytema et al., 1989 (**75**)). Sediment should be stored with no air over the sealed samples (no head space) at 4°C before the start of a test (Shuba et al., 1978 (**76**)). Sediment should be stored in containers constructed of suitable materials as outlined in 11.2.

11.6.4 Sediment cores collected for stratigraphical or geological studies can be stored at 4°C in a humidity-controlled room for several months without any substantial changes in sediment properties (Mudroch and Azcue, 1995 (**46**)).

12. Sample Manipulations

12.1 Manipulation of sediments in the laboratory is often required to achieve certain desired characteristics or forms of material for toxicity or bioaccumulation testing and chemical analysis. As all manipulation procedures alter some qualities of field samples, it is important to evaluate the effect that these changes might have on the study objective and on each measurement. Therefore, all procedures used to prepare sediment samples should be described in the study plan and documented. Generally, manipulation procedures should be designed to maintain sample representativeness in terms of toxicity and chemistry by minimizing procedural artifacts.

12.1.1 This section discusses methods for several common manipulations performed in the laboratory including sieving, spiking, organic carbon modification and formulated sediments, sediment dilution, and elutriate preparation. Other sediment manipulations, such as salinity adjustments or pretreatment of sediment ammonia (done in conjunction with toxicity testing in certain regulatory programs) are not discussed in this standard as these are described elsewhere (for example, PSEP, 1995 (**77**), USEPA 1994 (**78**)).

12.2 *Sieving:*

12.2.1 In general, sieving should not be done on sediment samples because this process can change the physicochemical characteristics of the sediment sample. For example, wet sieving of sediment through fine mesh (=500 µm openings) has been shown to result in decreased percent total organic carbon and decreased concentrations of total PCBs, which might have been associated with fine suspended organic matter lost during the sieving process (Day et al. 1995 (**79**)). Sieving can also disrupt the natural chemical equilibrium by homogenizing or otherwise changing the biological activity within the sediment (Environment Canada, 1994 (**2**); Test Method E1706).

12.2.2 In some cases, however, sieving might be necessary to remove indigenous organisms, which can interfere with subsequent toxicity testing and confound interpretations of analytical results (USEPA, 1994 (**78**); 2000d(**30**); Practice D3976). Indigenous organisms can be problematic in toxicity testing because they may be the same species as the test organism, they may be a species similar in appearance to test organisms, or they might prey on the test organisms. Similarly, in bioaccumulation tests, indigenous organisms might be similar in appearance to the test organisms (Test Method E1706 and Guide E1688).

12.2.3 If sieving is performed, it should be done for all samples to be tested, including control and reference sediments, if the objective of the study is to compare results among stations (Test Method E1706). It might be desirable to obtain certain measurements (for example, dissolved and total organic carbon, acid volatile sulfide [AVS], and simultaneously extracted metals [SEM]) both before and after manipulation, to document changes associated with sieving (USEPA, 2000d (**30**)). In addition, it might be desirable to document the effect of sieving on the sediment sample by conducting comparative toxicity tests using sieved and unsieved sediment (Environment Canada, 1994 (**2**)).

12.2.4 *Sieving Methods:*

12.2.4.1 *Press Sieving*—If sieving is necessary, press sieving is the preferred method. In this method, sediment particles are hand-pressed through a sieve using chemically inert paddles (Giesy et al. 1990 (**80**) ; Johns et al. 1991 (**81**)). Matter retained by the screen, such as organisms, shell fragments, gravel, and debris, should be recorded in a log book and discarded (USEPA/USACE, 1991 (**33**)). Samples with high debris, vegetation, or clay content might be difficult to press through a single sieve with a mesh size less than 1 mm; such samples might need to be pressed through a series of sieves with progressively smaller openings. Water should not be added to sediment when press sieving, as this could result in changes in contaminant concentration and bioavailability. Samples that are going to be used for both chemical analysis and toxicity or bioaccumulation tests should be sieved together, homogenized, and then split for their respective analyses.

12.2.4.2 *Wet Sieving*—If sediments cannot be hand-pressed sieved , wet sieving might be required, however, this type of sieving increases the likelihood of contaminant loss. Wet sieving involves swirling sediment particles within a sieve using water to facilitate the mechanical separation of smaller from larger particles. A slurry made with water that has separated from the sediment during storage or transport might be sufficient to wash particles through the sieve. Wet samples that might have settled during transit should be stirred to incorporate as much field water as possible. In some cases, addition of a small volume of site water, deionized water, or reconsitituted water to the wet sample might be required. Mechanical shakers or stirring with a nylon brush can also facilitate wet sieving (Mudroch and MacKnight, 1994 (**36**)).

12.2.4.3 In general, smaller mesh sieves are preferred to reduce loss of fines. Sieves made of stainless steel, or plastic woven polymers (for example, polyethylene, polypropylene, nylon, and polytetrafluoroethylene (PTFE)) with mesh sizes that vary from 0.24 to 2.0 mm have been used to sieve sediment for toxicity tests (Keilty et al. 1988a;b; [illegible]);

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