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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:

[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.

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[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**

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.

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

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 requirements.

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

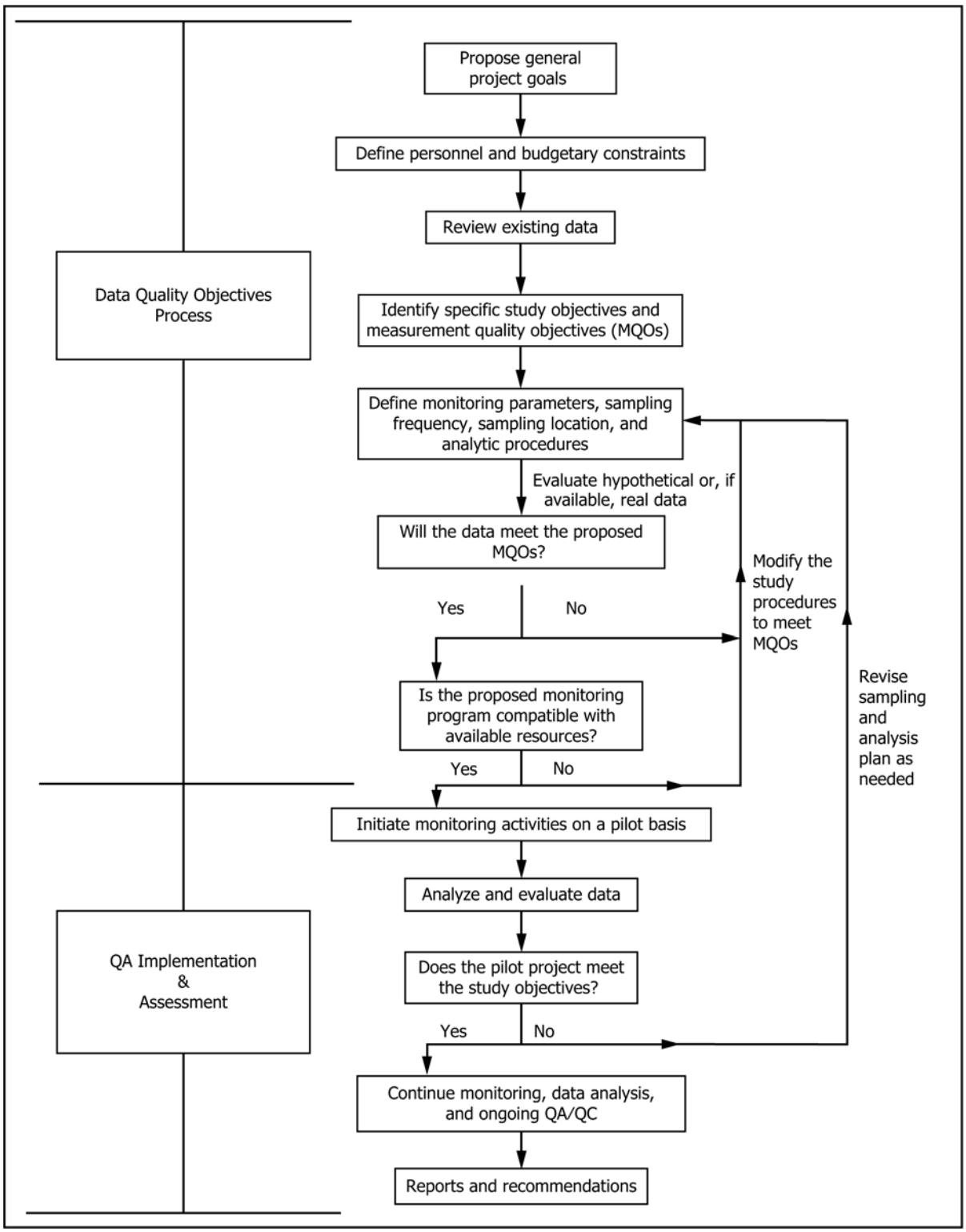


FIG. 1 Flow Chart Summarizing the Process that Should Be Implemented in Designing and Performing a Monitoring Study (modified from MacDonald et al. (1991 (10)); USEPA 2001 (1))

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

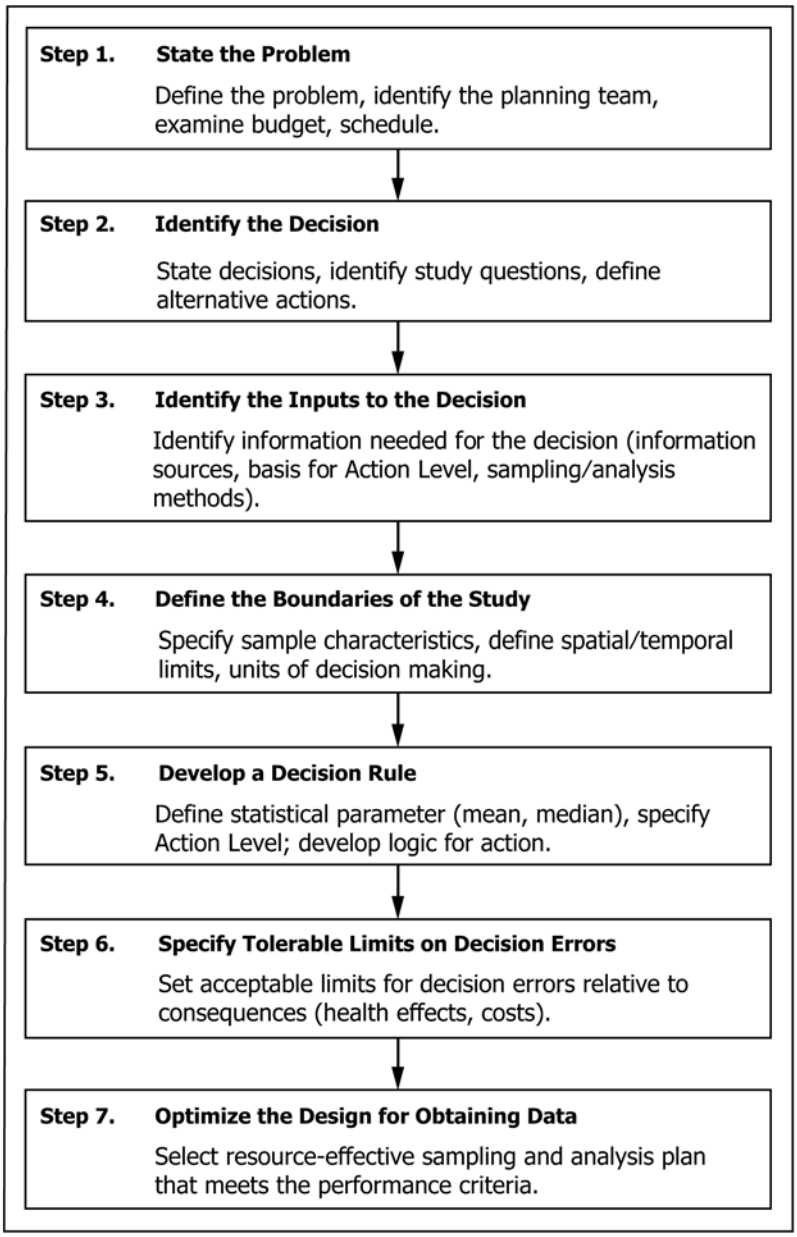


FIG. 2 Flow Chart Summarizing the Data Quality Objectives Process (after USEPA 2000a (12); 2001 (1))

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.

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

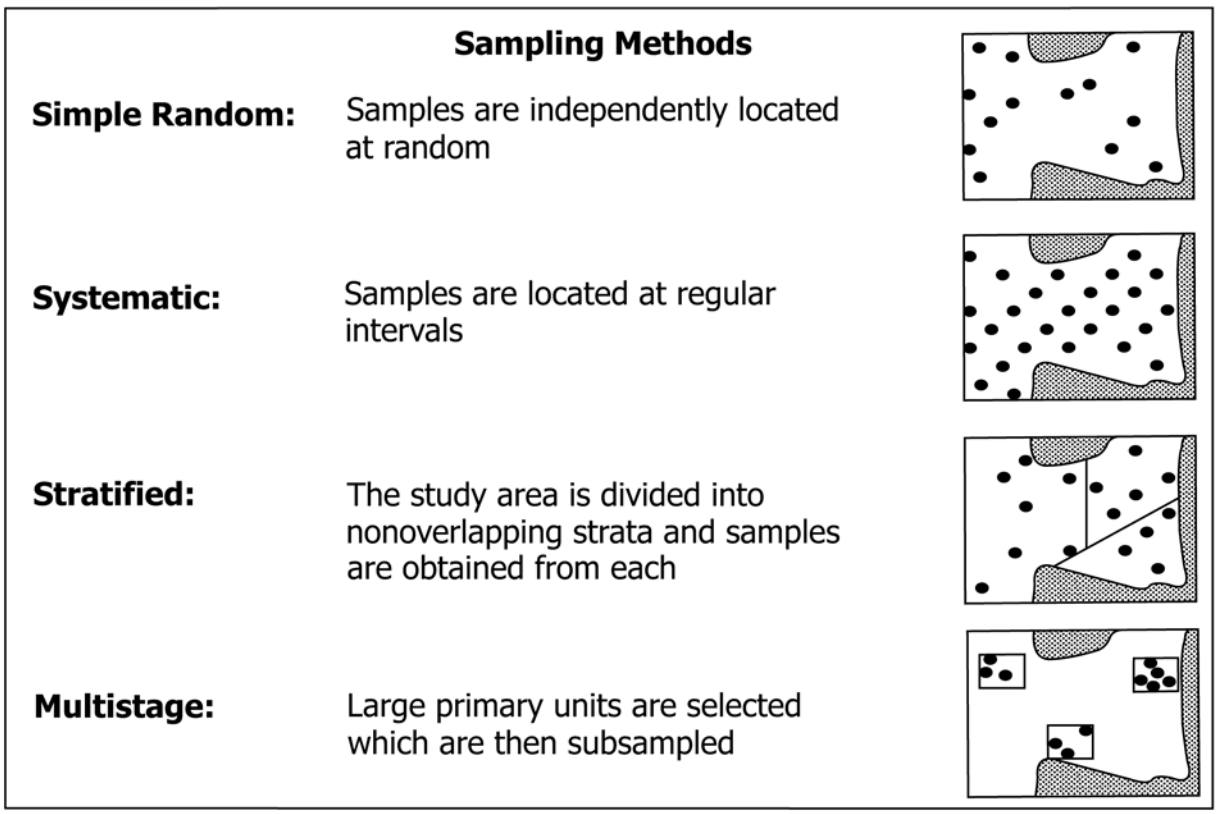


FIG. 3 Description of Various Sampling Methods (adapted from USEPA 2000c (20); 2001(1))

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.

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 10 to 20 cm of

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

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

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:*

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:*

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

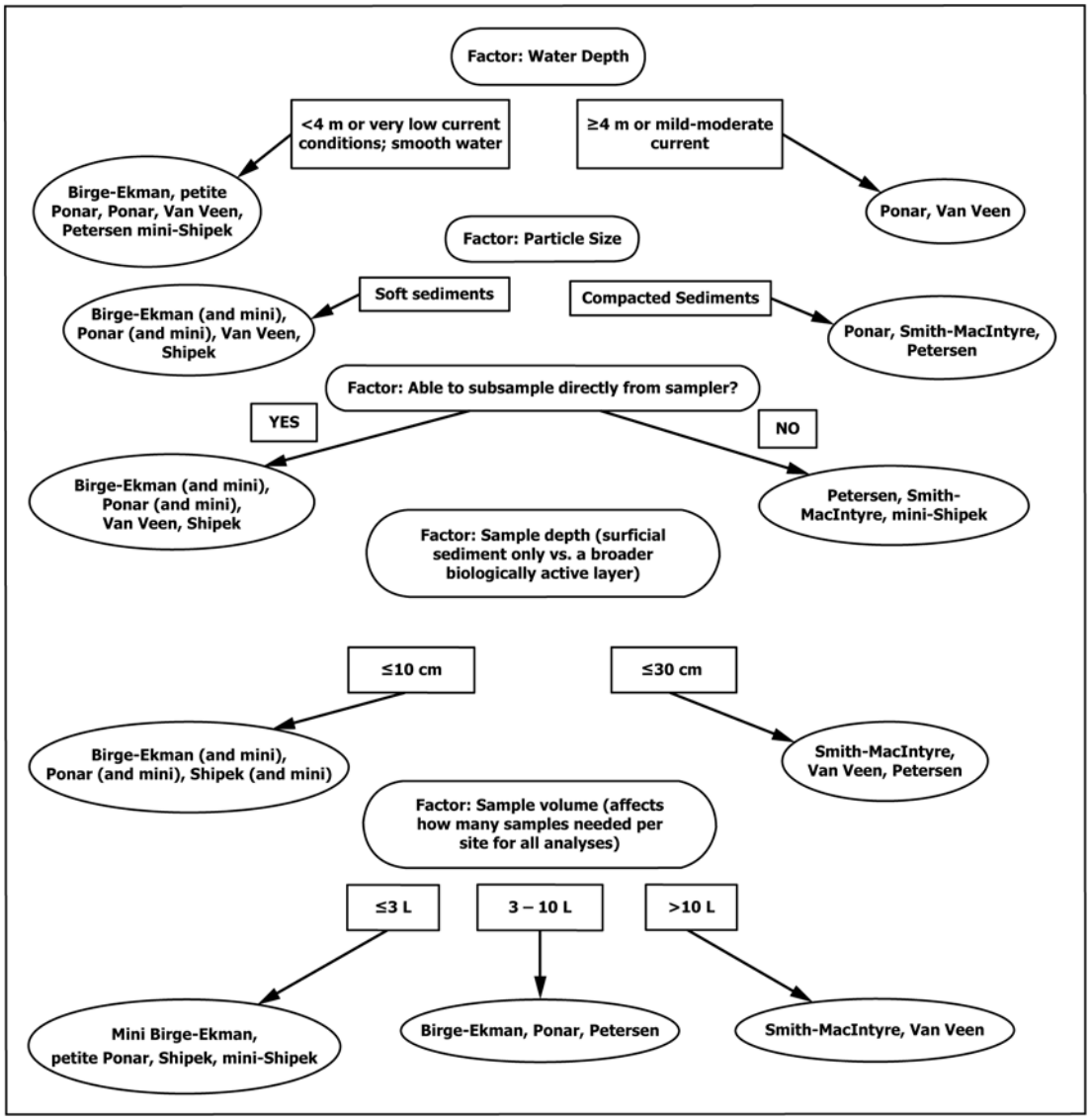


FIG. 4 Flowchart for Selecting Appropriate Grab Samplers Based on Site Specific or Design Factors (USEPA 2001 (1))

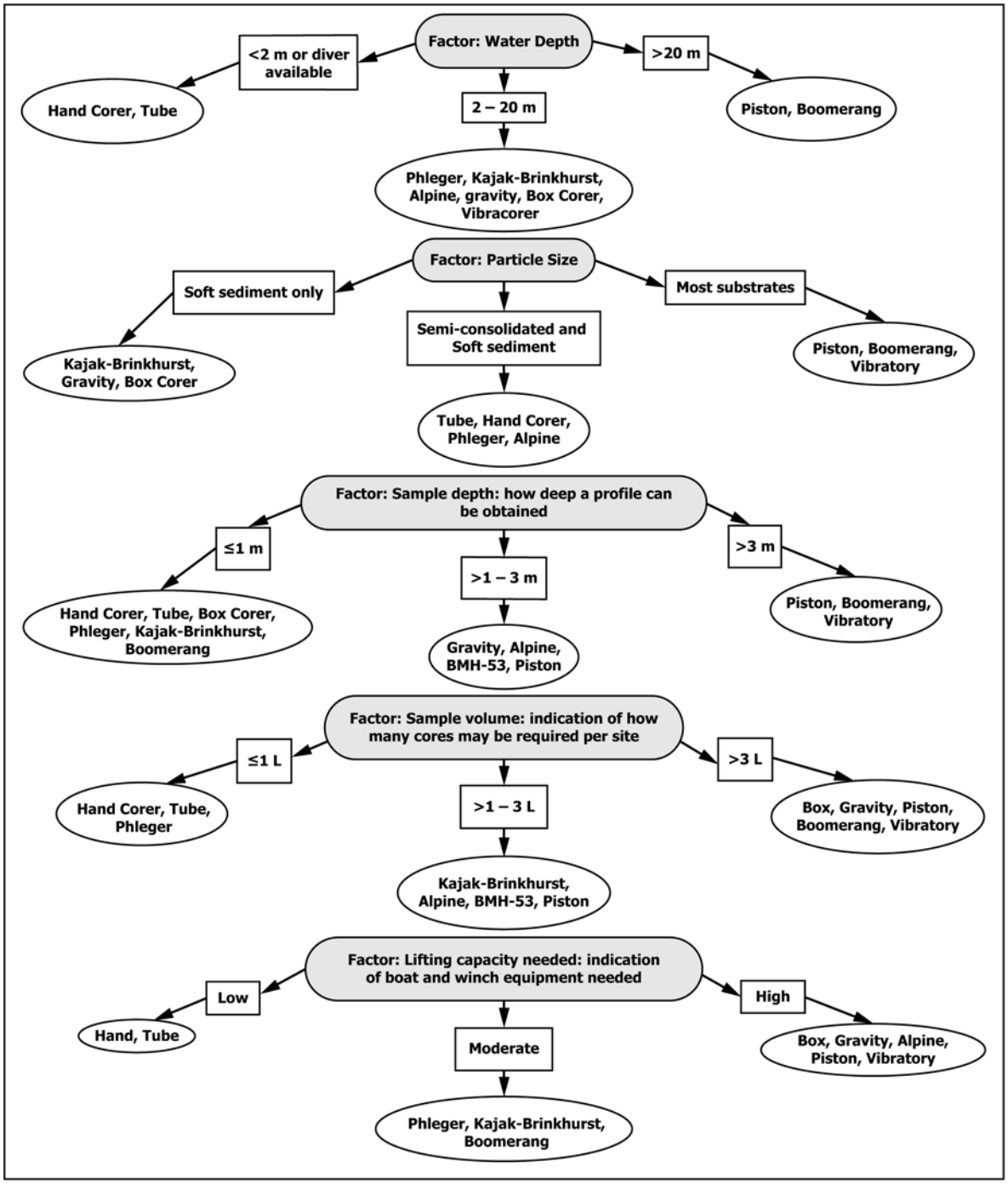


FIG. 5 Flowchart for Selecting Appropriate Core Samplers Based on Site-specific Factors (USEPA 2001 (1))

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

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).

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,

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

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

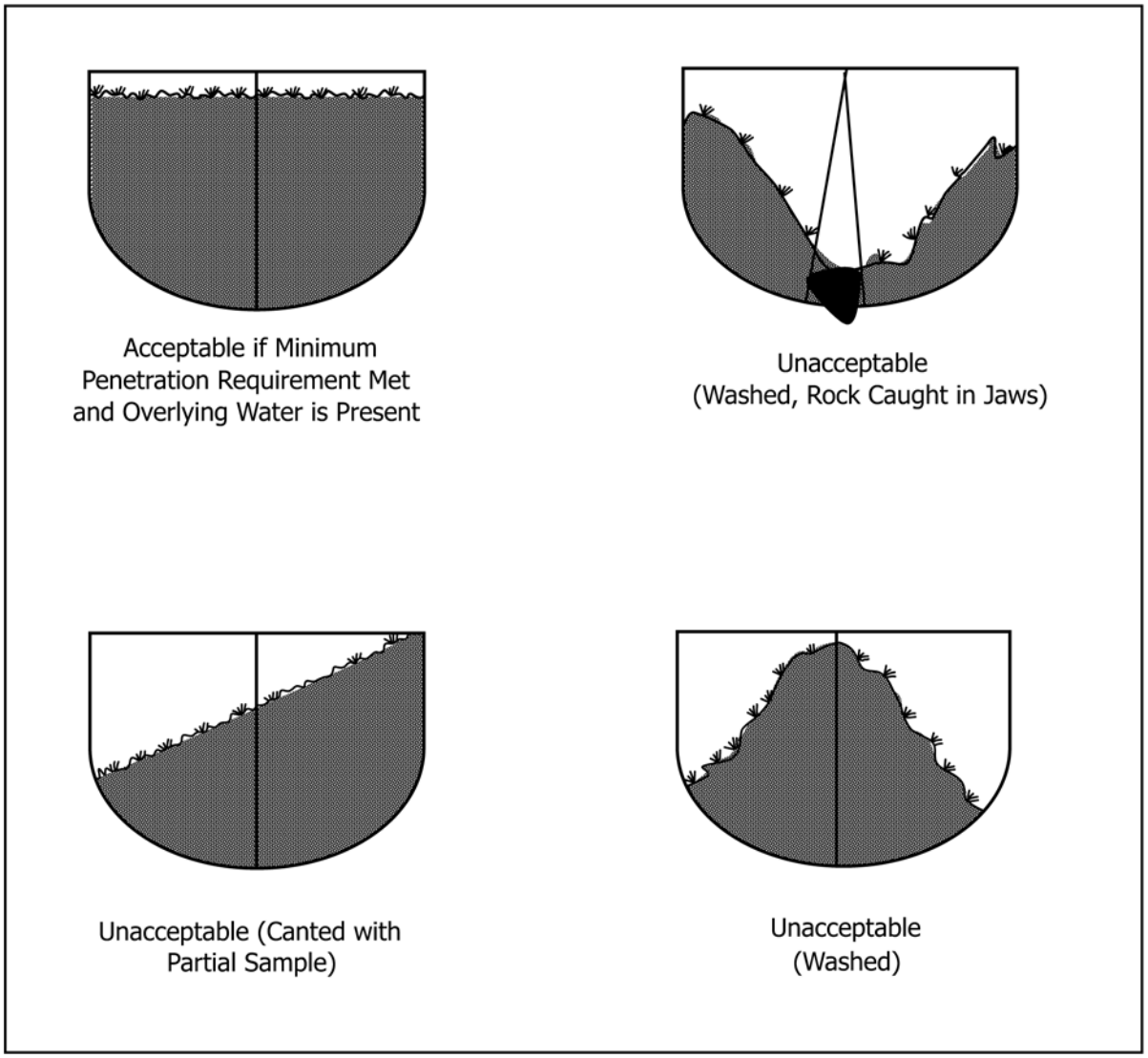


FIG. 6 Illustrations of Acceptable and Unacceptable Grab Samples (USEPA 2001 (1))

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,

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

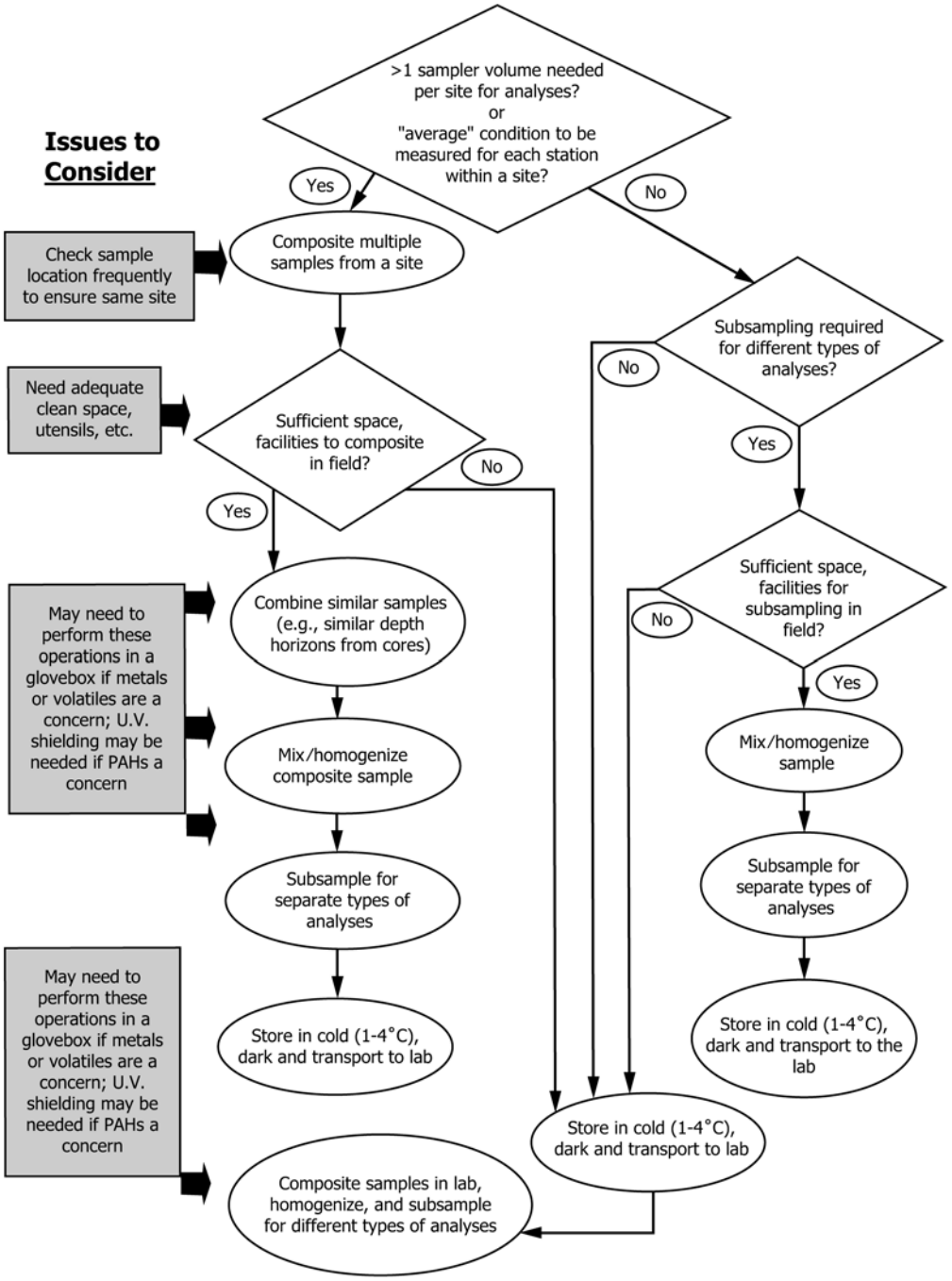


FIG. 7 Flowchart of Suggested Sediment Processing Procedures (USEPA 2001 (1))

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

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,

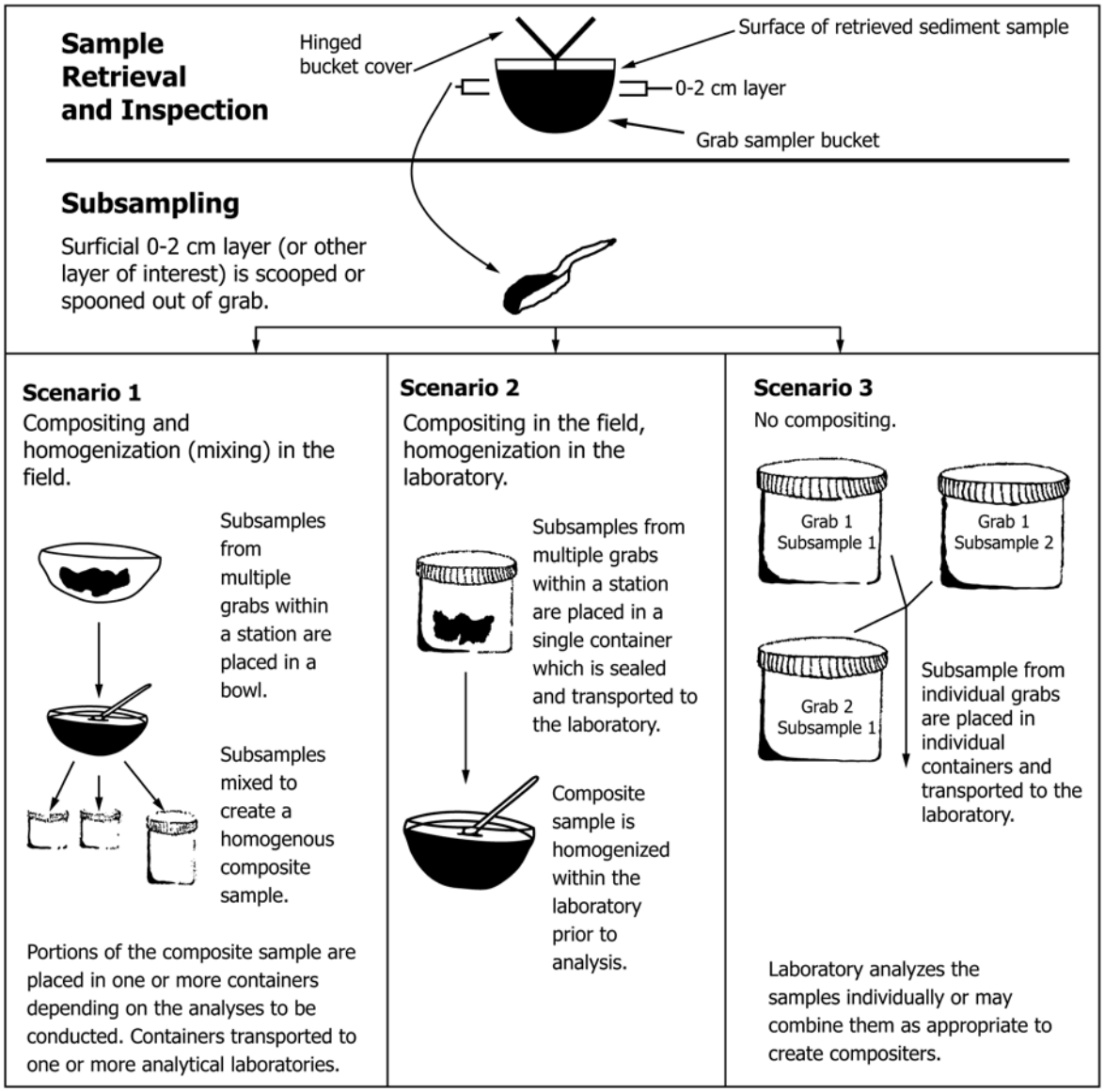


FIG. 8 Alternatives for Subsampling and Compositing Sediment Grab Samples (USEPA 2001(1))

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

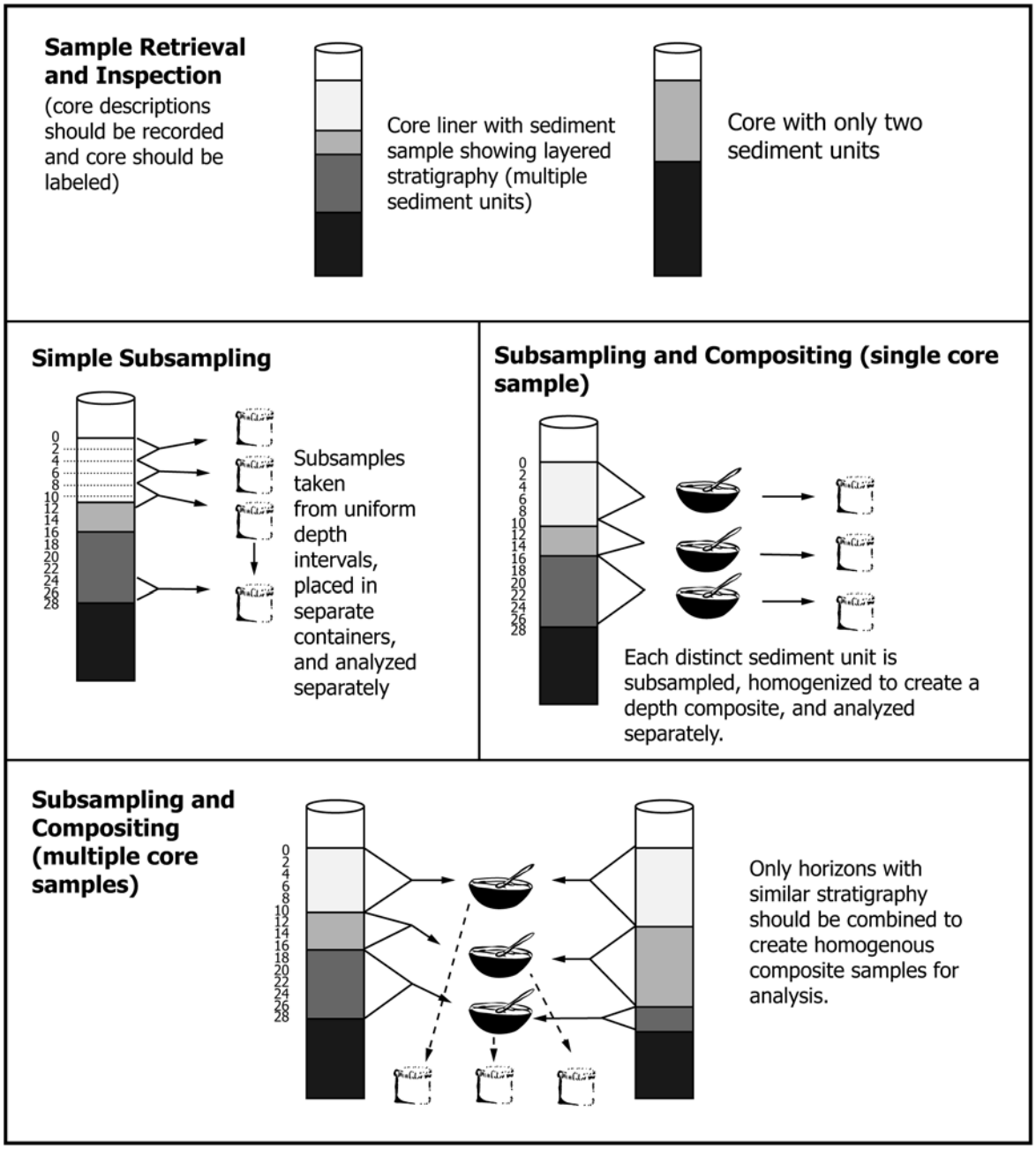


FIG. 9 Alternatives for Subsampling and Compositing Sediment Core Samples (USEPA 2001 (1))

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

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

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

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; (**82**),(**83**);

Giesy et al. 1990 (**80**); Lydy et al. 1990(**84**); Stemmer et al. 1990a;b (**61**), (**62**); Johns et al. 1991 (**81**); Landrum and Faust, 1991 (**85**)). Non-metallic sieves are preferred if metals are of interest. Stainless steel sieves are acceptable if organic compounds are of interest. Stainless steel (provided the mesh is not soldered or welded to the frame), nylon, or Nitex-type plastic sieves should be used when other inorganic constituents are of concern or are to be analyzed (PSEP 1995) (**77**).

12.2.4.4 Generally, sieving through a 10-mesh (2-mm openings) sieve is acceptable as a basis to discriminate between sediment and other materials. For toxicity testing, a mesh size of 1.0 mm has been used (Environment Canada, 1994 (**2**)) which will remove most adult amphipods. However, a mesh of 0.25 mm might be needed to remove immature amphipods and most macrofauna (Landrum et al. 1992 (**86**); Robinson et al. 1988 (**87**); Day et al. 1995 (**79**)). In marine sediments, sieves with a mesh size of 0.5 mm are effective in removing most of the immature amphipods (Swartz et al. 1990 (**88**); PSEP, 1995 (**77**)).

12.2.5 *Alternatives to Sieving*—Unwanted materials (for example, large particles, trash, and indigenous organisms), can be removed from the sediment sample using forceps, before or, as an alternative to, sieving. If anaerobic integrity of the sample is not a concern, the sediment could be spread on a sorting tray made of cleaned, chemically-inert material, and should be hand-picked with forceps. A stereomicroscope or magnifying lens might facilitate the process, or may be used to determine if sieving is necessary. Hand-picking is preferable to sieving because it is less disruptive, but it typically is not practical for large volumes of sediment. This process may oxidize the sediment and might alter contaminant bioavailability. Autoclaving, freezing, and gamma irradiation of sediments are alternatives to physical removal for inhibiting endemic biological activity in field-collected sediments. These are not generally recommended procedures. Each method has unique effects on the physicochemical and biological characteristics of the sediment, and a careful evaluation with respect to the study objectives is warranted when these methods are considered.

12.3 *Formulated Sediment and Organic Carbon Modification:*

12.3.1 *Formulated Sediments*—Formulated sediments (also called reconstituted, artificial, or synthetic sediments) are mixtures of materials that mimic the physical components of natural sediments (Test Method E1706). While they have not been used routinely, formulated sediments potentially offer advantages over natural sediments for use in chemical fate and biological effects testing. However, formulated sediments also have limitations. They do not possess the natural microbial, meiofaunal, and macrofaunal communities or the complex organic and inorganic gradients prevalent in natural sediments. The lack of biological activity, diagenesis, and oxidation-reduction (redox) potential gradients undoubtedly alters some sorption and desorption properties, which might in turn alter contaminant fate and effects. The current lack of understanding of physicochemical controls on bioavailability in different sediment environments precludes broad-scale use of formulated sediments (Test Method E1706).

12.3.2 A formulated sediment should: (*1*) support the survival, growth, or reproduction of a variety of benthic invertebrates, (*2*) provide consistent acceptable biological endpoints for a variety of species, and (*3*) be composed of materials that have consistent characteristics (USEPA, 2000d (**30**), Test Method E1706). Characteristics should include: (*1*) consistency of materials from batch to batch, (*2*) contaminant concentrations below concentrations of concern, and (*3*) availability to all individuals and facilities (Kemble et al. 1999 (**89**)). Physicochemical characteristics that might be considered when evaluating the appropriateness of a sediment formulation include percent sand/clay/silt, organic carbon content, cation exchange capacity (CEC), redox potential, pH, and carbon:nitrogen:phosphorous ratios (USEPA, 2000d (**30**); Test Method E1706).

12.3.3 The specific material source should be carefully selected, as characteristics can vary significantly among product types. For example, USEPA (2000d (**30**)) found that for three different sources of kaolinite clay, the percentage of clay ranged from 57 to 89 %, depending on individual product specifications. There are a number of suppliers of various sediment components (USEPA, 2000d (**30**)). A critical component of formulated sediments is the source of organic carbon. It is not clear that any one source of organic carbon is routinely superior to another source (Test Method E1706).

12.3.4 *Organic Carbon Modification*—Organic carbon content of natural as well as formulated sediments can be modified to assess the effect on contaminant fate and bioavailability. Many studies have modified sediment carbon because total organic carbon (TOC) content has been shown to be a major determinant of non-ionic organic chemical bioavailability (Di Toro et al. 1991 (**90**); DeWitt et al. 1992 (**91**); and Kosian et al. 1999 (**92**)). While TOC modifications might be necessary to achieve study objectives, it should be recognized that organic carbon manipulations can change the particle composition and size distribution, thereby potentially affecting contaminant equilibrium. Thus, results from such experiments should be interpreted with care. Also, the sample needs to be equilibrated (see 12.4.1) following addition of the new source of organic carbon, before conducting analyses.

12.3.5 Some recipes have used peat as the source of organic carbon, however, the quality and characteristics of peat moss can vary from bag to bag (Test Method E1706). Other sources of organic carbon include humus, potting soil, maple leaves, composted cow manure, rabbit chow, cereal leaves, chlorella, trout chow, Tetramin®, Tetrafin®, and alpha cellulose. Of these, only peat, humus, potting soil, composted cow manure, and alpha cellulose have been used successfully in sediment testing without fouling the overlying water; other sources have caused dissolved oxygen concentrations to fall to unacceptable levels (Kemble et al. 1999 (**89**)).

12.3.6 Five studies compared organic carbon sources in formulated sediments. A study of 31 different organic carbon recipes by Environment Canada (1995) (**93**) compared effects on sediment homogeneity, density, and turbidity. Cerophyll and trout chow were selected as the optimal organic carbon sources with high clay (kaolin at 50 or 75 % total concentration) and fine sand.

12.3.7 Ribeiro et al. (1994) (**94**) suggested the use of synthetic alpha-cellulose as a carbon source amended with humic acid. The use of alpha-cellulose in formulated sediment has since been evaluated by Kemble et al. (1999 (**89**), Sawyer and Burton (1994 (**95**), and Fleming and Nixon (1996 (**96**)). Ribeiro et al. (1994 (**94**)) found that sorption was dependent on the amount of organic carbon present. Kemble et al. (1999 (**89**)) found that growth of Hyalella azteca was better in 10 % than in 2 % alpha-cellulose. Both alpha-cellulose and conditioned red maple leaves were found to be suitable as organic carbon amendments for reference toxicant testing with Hyalella azteca (96 h exposures) when spiked with cadmium, zinc, or anthracene (Sawyer and Burton, 1994 (**95**)).

12.3.8 Use of alpha cellulose as a carbon source for sediment-spiking studies has not been adequately evaluated, but it appears to be promising. Alpha cellulose is a consistent source of organic carbon that is relatively biologically inactive and low in concentrations of chemicals of concern. Furthermore, Kemble et al. (1999 (**89**)) reported that conditioning of formulated sediment was not necessary when alpha cellulose was used as a carbon source for a negative control sediment. Compared with other sources of organic carbon, alpha cellulose is highly polymerized and would not serve as a food source, but rather would serve to add texture or provide a partitioning compartment for chemicals. Reductions in organic carbon content have been achieved by diluting sediment with clean sand (see 12.5; Clark et al. 1986 (**97**); Clark et al. 1987 (**98**); Tatem, 1986(**99**); Knezovich and Harrison, 1988) (**100**)). However, this can change sediment characteristics resulting in non-linear responses in toxicity (Nelson et al. 1993 (**101**)). Combustion has also been used to remove fractions of organic carbon (Adams et al. 1985 (**102**); IJC, 1988 (**103**)). However, this method results in substantial modification of the sediment characteristics, including oxidization of some inorganic components.

12.3.9 The ratio of carbon to nitrogen to phosphorous might be an important parameter to consider when selecting an organic carbon source. This ratio can vary widely among carbon sources (Test Method E1706, USEPA 2000d(**30**)). For example, carbon can range from 30 to 47 %, nitrogen from 0.7 to 45 mg/g, and phosphorous from below detection limits to 11 µg/g for several different carbon sources (USEPA, 2000d(**30**)).

12.3.10 A variety of formulations have been used successfully in sediment toxicity testing (Test Method E1706 and USEPA 2000d(**30**)). At this time, no one formulation appears to be universally better than others.

12.4 *Sediment Spiking:*

12.4.1 Test sediment can be prepared by manipulating the properties of a control or reference sediment (Test Method E1706). Mixing time (Stemmer 1990a, 1990b (**61**) (**62**)) and aging (Landrum 1989, Word et al, 1987, Landrum and Faust 1992(**104**),(**105**),(**106**)) of spiked sediment can affect bioavailability of chemicals in sediment. Many studies with spiked sediment are often started only a few days after the chemical has been added to the sediment. This short time period may not be long enough for sediments to equilibrate with the spiked chemicals. Consistent spiking procedures should be followed in order to make interlaboratory comparisons. Limited studies have been conducted comparing appropriate methods for spiking chemicals in sediment. Additional research is needed before more definitive recommendations for spiking of sediment can be outlined in this standard. The guidance provided in the following sections has been developed from a variety of sources. Spiking procedures that have been developed using one sediment or test organism may not be applicable to other sediments or test organisms.

12.4.2 Spiking involves adding one or more chemicals to sediment for either experimental or quality control purposes. Spiking environmental samples is used to document recoveries of an analyte and thereby analytical bias. Spiked sediments are used in toxicity tests to determine effects of material(s) on test species. The cause of sediment toxicity and the interactive effects of chemicals can be determined by spiking a sediment with chemicals or complex waste mixtures (**97**). Sediments spiked with a range of concentrations can be used to generate either point estimates (for example, LC50) or a minimum concentration at which effects are observed (lowest-observable-effect concentration; LOEC). Results of tests may be reported in terms of a Biota-sediment accumulation factor (BSAF) Ankley et al., 1992b (**107**). The influence of sediment physico-chemical characteristics on chemical toxicity can also be determined with sediment-spiking studies Swartz et al., 1994(**108**). Spiking tests can also provide information concerning chemical interactions and transformation rates. The design of spiking experiments, and interpretation of results, should always consider the ability of the sediment to sequester contaminants, recognizing that this governs many chemical and biological processes (O'Donnel et al. 1985 (**109**); Stemmer et al. 1990a,b (**61**),(**62**); Northcott and Jones, 2000 (**110**), Test Method E1706). In preparation for toxicity and bioaccumulation tests, references regarding the choice of test concentrations should be consulted (USEPA 2000d (**30**), Environment Canada 1995 (**93**), Test Method E1706). Table 12 summarizes general recommendations for spiking sediments with a chemical or other test materials.

TABLE 12 Recommendations for How to Spike a Sediment With a Chemical or Other Test Material (USEPA 2001 (1))

Regardless of the spiking technique used, care should be taken to ensure complete and homogenous mixing.
Replicate subsamples should be analyzed to confirm homogeneous mixing.
Moisture content should be determined on triplicates for each sample so that the spike concentration can be normalized on a dry weight basis.
Wet spiking is recommended over dry spiking methods.
Generally speaking, the jar rolling method is more suitable than hand mixing for spiking larger batches of sediment.
To ensure chemical equilibrium between the sediment and pore water in toxicity testing, spike sediments should be stored for at least one month, unless other information is available for the spiking material and sediment type.
Direct addition of organic solvent carriers should be avoided because they might alter sediment chemistry and affect contaminant bioavailability. Shell coating methods should be used instead as this eliminates many of the disadvantages of solvent carriers.

12.4.3 Several issues regarding sediment spiking are addressed in this section. First, several methods have been used to spike sediments but the appropriate method needs to be selected carefully depending on the type of material being spiked (for example, soluble in water or not), its physical-chemical form, and objectives of the particular study. Second, spiked material should be uniformly distributed throughout the sediment. Otherwise, chemical analyses, or toxicity or bioaccumulation tests, are likely to yield highly variable results, depending on the concentration of spiked material present. Third, the spiked material needs to be at equilibrium between the sediment and the interstitial water so that all relevant exposure phases are appropriately considered in chemical analyses or toxicity or bioaccumulation testing. The time it takes to reach this equilibrium is a critical factor that needs to be considered and documented.

12.4.4 The test material(s) should be at least reagent grade, unless a test using a formulated commercial product, technical-grade, or use-grade material is specifically needed. Before a test is started, the following should be known about the test material: (*1*) the identity and concentration of major ingredients and impurities, (*2*) water solubility in test water, (*3*) log Kow, BCF (from other test species), persistence, hydrolysis, and photolysis rates of the test substrate, (*4*) estimated toxicity to the test organism and to humans, (*5*) if the test concentration(s) are to be measured, the precision and bias of the analytical method at the planned concentration(s) of the test material, and (*6*) recommended handling and disposal procedures. Addition of test material(s) to sediment may be accomplished using various methods, such as a: (*1*) rolling mill, (*2*) feed mixer, or (*3*) hand mixing. Modifications of the mixing techniques might be necessary to allow time for a test material to equilibrate with the sediment. Mixing time of spiked sediment should be limited from minutes to a few hours, and temperature should be kept low to minimize potential changes in the physico-chemical and microbial characteristics of the sediment USEPA, 2000 (**111**). Duration of contact between the chemical and sediment can affect partitioning and bioavailability Word et al., 1987 (**105**). Care should be taken to evenly distributed the spiked material in the sediment. Analyses of sediment subsamples is advisable to determine the degree of mixing homogeneity Ditworth et al., 1990(**112**). Moreover, results from sediment-spiking studies should be compared with the response of test organisms to chemical concentrations in natural sediments (**113**).

12.4.5 Organic chemicals have been added: (*1*) directly in a dry (crystalline) form; (*2*) coated on the inside walls of the container (Ditsworth et al. (**112**)); or (*3*) coated onto silica sand (for example, 5 % w/w of sediment) which is added to the sediment (D.R. Mount, USEPA, Duluth, MN, personal communication). In techniques 2 and 3, the chemical is dissolved in solvent, placed in a glass spiking container (with or without sand), then the solvent is slowly evaporated. The advantage of these three approaches is that no solvent is introduced to the sediment, only the chemical being spiked. When testing spiked sediments, procedural blanks (sediments that have been handled in the same way, including solvent addition and evaporation, but contain no added chemical) should be tested in addition to regular negative controls. Metals are generally added in an aqueous solution (Di Toro et al. (**114**)). Ammonia has also been successfully spiked using aqueous solutions (Besser et al. (**115**)). Spiking blanks should also be included in these analyses.

12.4.6 Sufficient time should be allowed after spiking for the spiked chemical to equilibrate with sediment components. For organic chemicals, it is recommended that the sediment be aged at least one month before starting a test. Two months or more may be necessary for chemicals with a high log Kow (for example, >6; D.R. Mount, USEPA, Duluth, MN, personal communication). For metals, shorter aging times (1 to 2 weeks) may be sufficient. Periodic monitoring of chemical concentrations in pore water during sediment aging is highly recommended as a means to assess the equilibration of the spiked sediments. Monitoring of pore water during spiked sediment testing is also recommended.

12.4.7 If the test contains both a negative control and a solvent control, the survival, growth, or reproduction of the organisms tested should be compared in the two controls. If a statistically significant difference is detected between the two controls, only the solvent control may be used for meeting the acceptability of the test and as the basis for calculation of results. The negative control might provide additional information on the general health of the organisms tested. If no statistically significant difference is detected, the data from both controls should be used for meeting the acceptability of the test and as the basis for calculation of results (Guide E1241 and Test Method E1706). If performance in the solvent control is markedly different from that in the negative control, it is possible that the data are compromised by experimental artifacts and may not accurately reflect the toxicity of the chemical in natural sediments.

12.4.8 *Preparation for Spiking:*

12.4.8.1 Debris and indigenous organisms should be removed from sediment samples as soon as possible after collection to reduce deterioration of sediment quality due to decomposition of organic debris and dying infauna. If sediments are to be stored before spiking, they should be kept in sealed containers at 4°C.

12.4.8.2 Regardless of the spiking technique used, care should be taken to homogenize the sediment. Chemical analyses should be conducted to verify that concentrations of the spiked contaminants are uniform throughout the mixed material. Three or more subsamples of the spiked sediment should be randomly collected to determine the concentration of the substance being tested. In general, the coefficient of variation (CV) should be = 20 % for homogeneity of mixing to be considered sufficient (Northcott and Jones, 2000 (**110**)).

12.4.8.3 Temperatures should be kept cool during spiking preparation (for example, 4°C) due to rapid physicochemical and microbiological alterations which might occur in the sediment that, in turn, might alter bioavailability and toxicity (Test Method E1706, Environment Canada 1995 (**93**)). If spiking PAH compounds, it might be important to conduct spiking in the dark, or at least under low light as PAH toxicity has been shown to increase under ultraviolet light (Ankley et al. 1994 (**116**)).

12.4.8.4 A subsample of the spiked sediment should be analyzed for at least the following parameters: moisture content, pH, ammonia, total organic carbon (TOC), acid volatile sulfide (AVS), particle size distribution, and background levels of the chemical(s) to be spiked. Further characterization may include analyses of total volatile residue, pore water salinity (before and after any sieving), chemical oxygen demand, sediment oxygen demand, oxidation-reduction potential (Eh), metals, total chlorinated organic content, chlorinated organic compounds, and polycyclic aromatic hydrocarbons (see Section 15 for more information on physicochemical parameters often measured on sediments). It is particularly important to determine the TOC concentration if the sediment is to be spiked with a non-ionic organic compound, as organic carbon is the primary binding phase for such compounds (Di Toro et al. 1990 (**53**)). Similarly, the concentration of AVS (the primary binding phase for cationic metals in anoxic sediments) and TOC should be measured after spiking with a cationic metal (Ankley et al. 1996 (**54**); Leonard et al. 1999 (**117**)). The organic carbon composition may also be an important characteristic to determine in the sediment (for example, the C:N ratio; Landrum et al. 1997 (**118**)). Further, bioavailability may be more controlled by the desorption characteristics of the compound from sediment (for example, this can be measured by a Tenax[5] desorption method that appears to correlate well with bioaccumulation; Ten Hulscher et al. 2003 (**119**)).

12.4.8.5 The sediment moisture content measurement is used to calculate the amount of chemical spiked on a dry weight basis. Generally, the moisture content should be determined on triplicates for each sample by measuring the weight lost following 24 h of oven-drying at 105°C. After drying, the samples should be cooled to room temperature in a desiccator before taking dry weight measurements (Yee et al. 1992 (**120**)). The mean wet density, expressed as mg water/cm^3, is measured by using the same drying method on known sediment volumes. This allows spiking to be normalized from a volume basis to an equivalent dry weight basis.

12.4.9 *Methods for Spiking:*

12.4.9.1 Spiking of both wet and dry sediments is common, but wet spiking is preferable because drying might reduce the representativeness of the sample by changing its physicochemical characteristics. Methods differ mainly in the amount of water present in the mixture during spiking, the solvent used to apply the toxicant, and the method of mixing. Generally speaking, the jar rolling method is more suitable than hand mixing for spiking larger batches of sediment.

12.4.9.2 In addition to the above techniques, sediments may be spiked by hand stirring using a scoop or spatula, as long as the homogeneity of the mixture is verified. Eberbach and gyro-rotary shakers have also been used effectively to mix spiked sediments (Stemmer et al. 1990a (**61**)). Less commonly, chemical(s) are added to the water overlying the sediment and allowed to sorb with no mixing (Stephenson and Kane, 1984; (**121**) O'Neill et al. 1985 (**122**); Crossland and Wolff, 1985 (**123**); Pritchard et al. 1986 (**124**)).

12.4.9.3 *Sediment Rolling*—This sediment rolling technique requires a specific jar-rolling apparatus (for example, Ditsworth et al. 1990 (**60**)). Many other jar-rolling apparatuses are available, ranging in size and options available. This "rolling mill" method has been used to homogenize large volumes of sediments spiked with metals and non-ionic organic compounds. The primary disadvantage of this method is that the mixing apparatus needs be constructed or purchased. The jar-rolling apparatus used by Ditsworth et al. (1990 (**60**)) consists of eight parallel, horizontal rollers powered by an electric motor through a reduction gear, belts, and pulleys, which rotate cylindrical vessels containing the substrate mixtures. Mixing is accomplished gravimetrically by slowly rolling the jars (gallon-sized jars can be rolled at about 15 revolutions per minute). Optimally wetted, individual substrate particles adhere to each other and to the wall of the revolving jar until they cascade or tumble down the surface of the substrate mass. Water may be added to the substrate before rolling to adjust the sediment-to-water ratio for optimal mixing. If oxidation is a concern (for example, if the sample will be analyzed for metals), jar contents might need to be maintained in an inert atmosphere. If PAHs are of concern then jars should be shielded from light (Ankley et al. 1994 (**116**)).

12.4.9.4 Each jar should be loaded with the required amount of wet sediment (with a calculated mass of dry sediment required for the test) before introduction of the toxicant. Several 1-cm diameter holes of different depths can be punched into the sediment to provide more surface area for the initial distribution of the test material. A predetermined volume of the stock solution or a serial dilution of the stock should be used to spike each jar load of sediment. A volumetric pipette can be used to distribute each aliquot onto the top surface and into the holes of the sediment in each jar. Sediments should be spiked sequentially, proceeding from low to high concentrations of test material, to minimize cross-contamination. Control sediment should be prepared by adding an equivalent volume of water to a jar loaded with unspiked sediment. After spiking, all jars and their contents should be processed identically.

12.4.9.5 Typically, jars should be rolled for greater than two hours to achieve sample homogeneity. Jars should be closely monitored during the first hour of rolling in order to achieve proper mixing of substrates. After rolling for about 15 min, mixing efficiencies of the substrates can be judged visually. If a sediment displays excessive cohesiveness, as indicated by agglomerating or balling, the jars should be opened and an aliquot of water (for example, 50 mL of water) added to each substrate to increase the fluidity. This procedure should be repeated as necessary until the operator visually observes that all substrates are tumbling without forming balls. Adding water in small rather than large aliquots can prevent over-saturation of the sediment. Over-saturation is undesirable because excess water needs to be decanted following rolling, and before sediment testing.

12.4.9.6 After rolling, the jars should be gently shaken to settle sediment that adhered to the walls. They may be set upright and stored overnight in the dark at room temperature or at an alternate temperature (for example, 4°C) depending on the study objectives. After equilibration (see 12.4.10) and

[5] Tenax is a trademark of Tenax Corporation 4800 East Monument St. Baltimore, MD 21205 .

before distributing the sample to test chambers, additional rolling for two hours will help integrate interstitial water into the sediment.

12.4.9.7 *Sediment Suspension Spiking*—The sediment suspension technique (Cairns et al. 1984 (**125**); Schuytema et al. 1984 (**126**); Stemmer et al. 1990a; b (**61**), (**62**); Landrum and Faust, 1991(**85**); Landrum et al. 1992 (**86**)) is the simplest of the three spiking techniques and requires the least equipment. The method involves placing water and sediment together in a 1-L beaker. The desired amount of toxicant, dissolved in water, is added to the beaker. The mixture should be stirred at a moderate speed with a stir bar, or mechanical stirrer, for a minimum of four hours. The sediment in the beakers should then be allowed to settle and equilibrated at the appropriate test temperature as specified in the method. The excess water overlying the sediment is decanted and discarded, and the sediment is distributed to the test containers (Environment Canada, 1995 (**93**)).

12.4.9.8 *Slurry Spiking*—The slurry technique (Birge et al., 1987 (**127**); Francis et al., 1984 (**128**); Landrum and Faust, 1991 (**85**); Landrum et al., 1992 (**86**)) requires a minimum of equipment and involves less water than the sediment suspension technique. A 250-g dry weight sample of sediment is placed in a 500-mL Erlenmeyer flask. Via a 25-mL aliquot of distilled, deionized water, a sufficient concentration of the materials of interest is added to obtain the desired sediment concentration (mg/kg, dry weight basis). Control (unspiked) sediment receives a 25-mL aliquot of distilled, deionized water having no added materials. The sealed flask may be mixed using various methods such as continuous agitation in a shaker for five days (Birge et al. 1987 (**127**)) or vigorous shaking for 60 s, twice daily for seven days (Francis et al. 1984 (**128**)). Following mixing, the sediment suspensions should be centrifuged to remove water. The moisture content of the sediment should be about 15 to 20 % after centrifugation. After removal of excess water, the prepared sediment can be placed in the exposure chambers and covered with water according to the specific methods. This procedure often yields sediment having its original moisture content.

12.4.10 *Equilibration Time:*

12.4.10.1 Before distributing the spiked sediment to containers for toxicity or bioaccumulation testing, or chemical analyses, the spiked sediments should be stored for a sufficient time to approach chemical equilibrium in the test material between the sediment and interstitial water (see 12.4.6). Equilibration times for spiked sediments vary widely among studies (Burton, 1991 (**129**)), depending on the spiking material and sediment type. For metals, equilibration time can be as short as 24 h (Jenne and Zachara, 1984 (**130**); Nebeker et al. 1986 (**131**)), but one to two weeks is more typical (Test Method E1706). For organic compounds with low octanol-water partition coefficients (Kow), equilibration times as short as 24 h have been used (DeWitt et al. 1989 (**132**)). Some organic contaminants might undergo rapid microbiological degradation depending on the microbial population present in the sample. In these cases, knowledge of microbial effects might be important in defining an appropriate equilibration period. Organic compounds with a high partition coefficient might require two months or more to establish equilibrium (Landrum et al. 1992 (**86**)). Boundaries for the sorption time can be estimated from the partition coefficient, using calculations described by Karickhoff and Morris (1985a,b (**133**), (**134**)). It is important to recognize that the quantity of spiked chemical might exceed the capacity of the test sediment system, prohibiting equilibrium.

12.4.10.2 Unless definitive information is available regarding equilibration time for a given contaminant and sediment concentration, a one-month equilibration period is recommended, with consideration that two months might be needed in some instances (see 12.4.10, USEPA 2000d (**30**)). Periodic monitoring during the equilibration time is highly recommended to empirically establish stability of interstitial water concentrations (USEPA, 2000d (**30**)). Sediment and interstitial water chemical concentrations should also be monitored during long-term toxicity tests to determine the actual chemical concentrations to which test organisms are exposed, and to verify that the concentrations remain stable over the duration of the test.

12.4.11 *Use of Organic Solvents:*

12.4.11.1 Direct addition of organic solvents should be avoided if possible, because organic solvents can alter geochemistry and bioavailability (USEPA, 2000d (**30**)). However, many organic materials require use of a solvent to adequately mix with the sediment. If an organic solvent is to be used, the solvent should be at a concentration that does not affect test organisms and should be uniform across treatments. Further, both solvent control and negative control sediments should be included in tests with solvents. The solvent concentration in the control should equal the treatment concentration, and should be from the same batch used to make the stock solution (Test Method E1706).

12.4.11.2 Organic solvents such as triethylene glycol, methanol, ethanol, or acetone may be used, but they might affect TOC levels, introduce toxicity, alter the geochemical properties of the sediment, or stimulate undesirable growth of microorganisms. Acetone is highly volatile and might leave the system more readily than triethylene glycol, methanol, or ethanol. A surfactant should not be used in the preparation of a stock solution because it might affect the bioavailability, form, or toxicity of the test material.

12.4.11.3 To reduce the possibility of solvent-related artifacts, the spiking process should include a step which allows the solvent to evaporate before addition of sediment and water followed by rolling (McLeese et al. 1980 (**135**); Muir et al. 1982 (**136**); Adams et al. 1985 (**102**)). Highly volatile organic compounds have been spiked into sediments using co-solvents followed by shaking in an aqueous slurry. When highly volatile compounds are used, immediate testing in covered flow-through systems is recommended (Knezovich and Harrison, 1988 (**100**)).

12.4.11.4 There is some uncertainty concerning artifacts introduced by the use of solvents. The use of a polar, water soluble carrier such as methanol was found to have little effect on the partitioning of non-ionic compounds to dissolved organic matter at concentrations up to 15 % carrier by volume (Webster et al. 1990 (**137**)). However, another study showed

that changes in partitioning by a factor of about two might occur with 10 % methanol as a co-solvent for anthracene sorption (Nkedi-Kizza et al. 1985 (**138**)). The effect of carrier volume on partitioning of organic chemicals in sediments is equivocal. However, because solvents might be either directly or indirectly toxic to the test organisms, caution should be taken to minimize the amount of carrier used. In addition, the use of a carrier such as acetone might result in faster equilibration of spiked organic compounds (Schults et al. 1992 (**139**)).

12.4.11.5 Shell coating techniques which introduce dry chemical(s) to wet sediment have also been developed, principally to eliminate the potential disadvantages of solvent carriers. The chemical may be either coated on the inside walls of the container (Ditsworth et al. 1990 (**60**); Burgess et al. 2000 (**140**)) or coated onto silica sand (Kane-Driscoll and Landrum, 1997 (**141**); Cole et al. 2000 (**142**); see 12.4.5). In each shell coating method, the chemical is dissolved in solvent, placed in a glass spiking container (with or without sand), and the solvent is slowly evaporated before addition of the wet sediment. Wet sediment then sorbs the chemical from the dry surfaces. It is important that the solvent be allowed to evaporate before adding sediment or water.

12.5 *Preparation of Sediment Dilutions:*

12.5.1 Spiked or field-contaminated sediments can be diluted with whole sediment to obtain different contaminant concentrations for concentration-effects testing. The diluent sediment should have physicochemical characteristics similar to the test sediment, including organic carbon content and particle size, but should not contain concentrations of contaminants above background levels (Test Method E1706, Burton 1991 (**129**)). Diluent sediment has included formulated sediment as well as reference or control sediment. Diluted sediment samples should be homogenized and equilibrated in accordance with procedures described in 11.5 and 12.4.10.

12.5.2 The diluent sediment should be combined with the test sediment in ratios determined on a dry weight basis to achieve the desired nominal dilution series. Volume to volume dilutions have also been performed (for example, Schlekat et al. 1995 (**143**); Johns et al. 1985 (**144**)), but weight to weight dilutions are preferred because they provide more accurate control and enable a more straightforward calculation of dose-response curves.

12.5.3 Results from dilution experiments should be interpreted with care. There can be non-linear responses due to non-equilibrium, non-linear sorption-desorption processes that cannot always be adequately controlled (Nelson et al. 1993 (**101**)). Nelson et al. (1993) (**101**) found that analyses of diluted sediments did not match nominal concentrations as estimated by physical characteristics and suggested that chemical characterization is needed to determine effects of manipulations (that is, mixing) and resulting changes (that is, oxygenation of complexing agents such as acid volatile sulfides). Hayward (2003 (**145**)) successfully conducted sediment dilution studies with field-collected sediments by matching the physical characteristics of the sediments, and by including a prolonged (3 month) equilibration period of the diluted sediment before conducting toxicity testing in the laboratory or field-colonization studies.

12.6 *Preparation of Sediment Elutriates:*

12.6.1 Sediment toxicity studies have evaluated aqueous extractions of suspended sediment called elutriates. The elutriate method was initially developed to assess the effects of dredging operations on water quality (USACE, 1976 (**146**)). Elutriate manipulations are also applicable to any situation where the resuspension of sediment-bound toxicants is of concern, such as bioturbation and storms, and that might disturb sediments and affect water quality (USEPA/USACE, 1991(**33**), 1998 (**35**); Ankley et al. 1991 (**147**)). USEPA/USACE (1998) (**35**)lists eighteen freshwater, estuarine, or marine aquatic organisms as candidates for elutriate toxicity testing. Standard effluent toxicity test procedures are also appropriate for elutriates, including tests with various vascular and non-vascular plant species (Ingersoll, 1995 (**148**)).

12.6.2 Elutriate tests are not intended to reflect the toxicity of interstitial waters or whole sediments, as there are differences in contaminant bioavailability in the two types of media (Harkey et al. 1994 (**113**)). In general, elutriates have been found to be less toxic than bulk sediments or interstitial water fractions (Burgess et al. 1993 (**47**); Ankley et al. 1991 (**147**)), although in some studies elutriates have been found to be more toxic (Hoke et al. 1990 (**149**)) or equally as toxic (Flegel et al. 1994 (**150**)) relative to interstitial water.

12.6.3 While there are several procedural variations, the basic method for elutriate preparation involves combining various mixtures of water and sediment (usually in the ratio of 4 parts water to 1 part sediment, by volume) and shaking, bubbling or stirring the mixture for 1 h (Ross and Henebry, 1989 ; Daniels et al. 1989 (**151**); Ankley et al. 1991 (**147**); Burgess et al. 1993 (**47**); USEPA/USACE, 1991(**33**), 1998 (**35**)). It is likely that chemical concentrations will vary depending on the elutriate procedure used. The water phase is then separated from the sediment by settling or centrifugation. Once an elutriate has been prepared, it should be analyzed or used in biological tests immediately, or as soon as possible thereafter. It should be stored at 4°C for not longer than 24 h, unless the method dictates otherwise (Environment Canada, 1994 (**2**); USEPA/USACE, 1991 (**33**), 1998 (**35**)). For toxicity test exposures exceeding 24 h, fresh elutriate should be prepared daily.

12.6.4 Filtering the elutriate is generally discouraged, but it might be prescribed for some toxicity tests. Filtration can reduce the toxicity of sediment elutriates due to sorption of dissolved chemicals on the filtration membrane and retention of colloids. If colloidal material needs to be removed, serial or double centrifugation is generally a preferred alternative. If an elutriate is filtered, it is recommended that only pre-treated filters be used and that the first 10 to 15 mL of the elutriate to pass through the filter be discarded (Environment Canada, 1994 (**2**)). Testing with a filtered elutriate should include an assessment to determine the extent of analyte adsorption or desorption to or from the filter.

13. Collection of Interstitial Water

13.1 Sediment interstitial water, or pore water, is defined as the water occupying the spaces between sediment or soil particles (Terminology E943). Interstitial water might occupy about 50 % (or more) of the volume of a depositional (silt-clay) sediment. The interstitial water is in contact with sediment surfaces for relatively long periods of time and therefore, might become contaminated due to partitioning of the contaminants from the surrounding sediments. In addition, interstitial waters might reflect ground water - surface water transition zones in upwelling or downwelling areas. In these areas, their chemistry might be more reflective of ground or surface waters at the site. Therefore, flow, residence time, and other physicochemical factors (for example, pH, temperature, redox potential, organic carbon, sulfides, carbonates, mineralogy) might have varying roles in determining whether interstitial waters are contaminated.

13.1.1 In many depositional sediments, interstitial waters are relatively static, and therefore, contaminants in the interstitial water and in the solid phase are expected to be at thermodynamic equilibrium. This makes interstitial waters useful for assessing contaminant levels and associated toxicity. Interstitial water is often isolated to provide either a matrix for toxicity testing, or to provide an indication of the concentration or partitioning of contaminants within the sediment matrix.

13.2 *General Procedures:*

13.2.1 Interstitial water sampling has become especially important because interstitial water toxicity tests yield additional information not provided by whole-sediment elutriate or sediment extract tests (Carr and Chapman 1992 (**152**); SETAC 2003 (**153**)). Furthermore, interstitial water toxicity tests are useful in sediment toxicity identification evaluation (TIE) studies (for example, Burgess 1996 (**154**) ; Carr 1998 (**155**); Burton et al. 2001 (**156**)) as test procedures and sample manipulation techniques can be faster and easier to conduct than whole-sediment toxicity tests (SETAC, 2003 (**153**)). Thus, the collection of interstitial water has become increasingly important in sediment quality monitoring programs.

13.2.2 Interstitial water sampling is most suitable for sediment types ranging from sandy to uncompacted silt-clays (Sarda and Burton, 1995 (**157**); SETAC, 2003 (**153**)). Such sampling is not typically performed on sediments with coarse particle size (such as gravel) or on hard, compacted clays, as the potential for interstitial water contamination in these sediment types is relatively low.

13.2.3 As with all sampling discussed in this standard, the principle aim is to use procedures that minimize changes to the in situ condition of the water. It should be recognized that most sediment collection and processing methods have been shown to alter interstitial water chemistry (for example, Schults et al. 1992 (**139**); Bufflap and Allen, 1995 (**158**); Sarda and Burton, 1995 (**157**)), thereby potentially altering contaminant bioavailability and toxicity.

13.2.4 Laboratory-based methods (for example, centrifugation, pressurization, or suction) are commonly used as alternatives to in-situ interstitial water collection (see 13.3). While these methods have been shown to alter interstitial water chemistry, they are sometimes necessary or preferred, especially when larger sample volumes are required (for example, for toxicity testing).

13.2.5 Both in-situ and laboratory-based or ex-situ methods might be appropriate for many study objectives. It is important that the same procedures are used for all stations sampled in a study so that appropriate comparisons can be made. Furthermore, the sediment depth at which interstitial water is sampled (either using in-situ or ex-situ extraction methods) should match the depth of interest in the study (see 10.1, SETAC 2003 (**153**)). For example, samples for dredging remediation should be sampled to the depth to be disturbed by dredging activity, whereas samples for a status and trends survey should be collected at the biologically active depth (often <15 cm). Fig. 10 summarizes the major considerations for selecting in-situ or ex-situ procedures in a given study.

13.2.6 The two major issues of concern regarding interstitial water sample integrity are: (*1*) the ability of the sampling device to maintain physicochemical conditions in the natural state by minimizing adsorption or leaching of chemicals to or from the device, and (*2*) the ability to maintain the sample in the redox state existing at the site. Precautions required to reduce sample artifacts will vary with each study as indicated in the following sections.

13.3 *In-situ Collection:*

13.3.1 In situ methods might be superior to ex-situ methods for collecting interstitial water, as they are less subject to sampling or extraction related artifacts and therefore, might be more likely to maintain the chemical integrity of the sample (Sarda and Burton 1995 (**157**), SETAC 2003 (**153**)). However, in situ methods have generally produced relatively small volumes of interstitial water, and are often limited to wadeable or diver-accessible water depths. These logistical constraints have limited their use and applicability in sediment monitoring studies.

13.3.2 The principal methods for in situ collection of interstitial water involve either deployed “peepers” (Bufflap and Allen, 1995 (**158**); Brumbaugh et al. 1994 (**27**); Adams, 1991 (**159**); Carignan and Lean, 1991 (**160**); Carignan et al. 1985 (**161**); Bottomley and Bayly, 1984 (**162**)) or suction techniques (Watson and Frickers, 1990 (**163**); Knezovich and Harrison, 1988 (**100**); Howes et al. 1985 (**164**)). A summary of these methods is provided in Table 13. Both methods have a high likelihood of maintaining in situ conditions. In cases where in situ deployment is impractical, peepers or suction devices can be placed in relatively undisturbed sediments collected by core or grab samplers (see Section 10).

13.3.3 *Peeper Methods:*

13.3.3.1 Peepers are small chambers with membrane or mesh walls containing either distilled water or clean water of the appropriate salinity or hardness. Samples are collected by burying the devices in sediments and allowing surrounding interstitial waters to infiltrate. In principle, dissolved solutes will diffuse through the porous wall into the peeper and the contained water will reach equilibrium with the ambient interstitial water. The design concept for sediment peepers originated as modifications of the dialysis bag technique used by Mayer (1976 (**165**)) and Hesslein (1976 (**166**)), and has

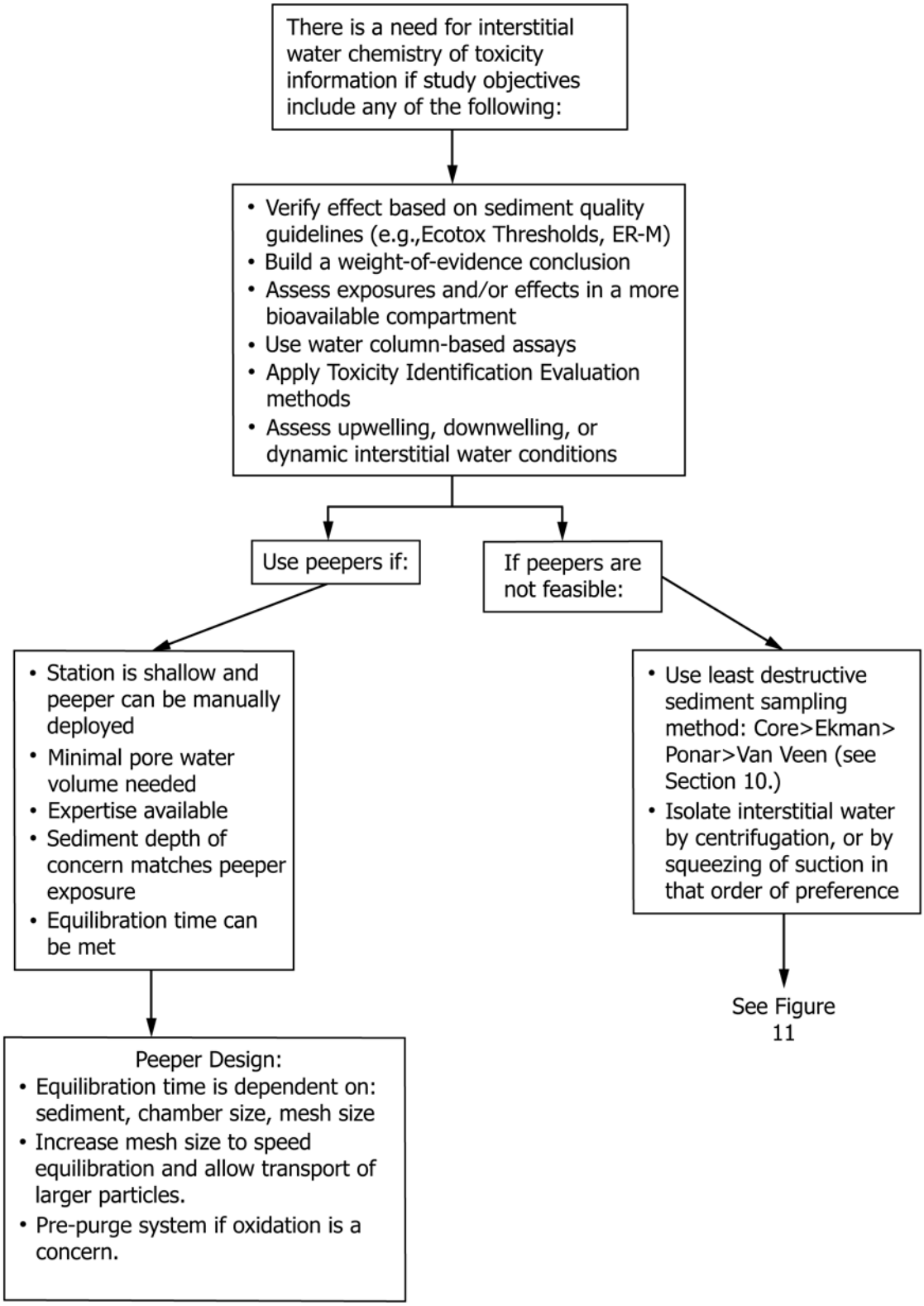


FIG. 10 Considerations for Selecting the Appropriate Type of Interstitial Water Sampling Method (USEPA 2001 (1))

been modified for use in laboratory sediment toxicity tests (Doig and Liber, 2000 (**167**)). The initial designs consisted of either a flat base plate or a cylindrical dialysis probe (Bottomley and Bayly, 1984 (**162**)) with compartments covered by dialysis membranes and a manifold for collection of multiple samples at various depths in the sediment profile Fig. 11. Further modifications to these designs have incorporated sampling ports, large sample compartments, and various types of

TABLE 13 In-situ Interstitial Water Collection Methods (Sarda and Burton 1995(157), SETAC 2003 (153))

NOTE 1—Incorporation of filtration into any collection method might result in loss of metal and organic compounds.

Device	Sediment Depth, cm	Sample Volume, L^3	Advantages	Disadvantages
Peeper	0.2 to 10	≤ 0.5	Most accurate method, reduced artifacts, no lab processing; relatively free of effects from temperature, oxidation, and pressure; inexpensive and easy to construct; some selectivity possible depending on nature of sample via specific membranes; wide range of membrane/mesh pore sizes, and/or internal solutes or substrates available.	Requires deployment by hand, thus requiring diving in >0.6 m depth water; requires hours to days for equilibration (varies with site and chamber); some membranes such as dialysis/cellulose are subject to biofouling; must deoxygenate chamber and materials to prevent oxidation effects; some construction materials yield chemical artifacts; some chambers only allow small sample volumes; care must be used on collection to prevent sample oxidation.
In situ Suction	0.2 to 30	≤ 0.25	Reduced artifacts, gradient definition; rapid collection, no lab processing; closed system which prevents contamination; methods include airstone, syringes, probes, and core-type samplers.	Requires custom, non-standard collection devices; small volumes; limited to softer sediments; core airstone method; difficult in some sediments and in deeper water (> 1 m); method might require diving for deployment in deep waters; methods used infrequently and by limited number of laboratories.

membranes with different pore sizes. These modifications are usually required based on specific project objectives regarding sample volumes and contaminants of interest.

13.3.3.2 Various peeper devices have been recently used effectively to collect interstitial water. For example, a simplified design using a 1 µm polycarbonate membrane over the opening of a polyethylene vial was successful in capturing elevated levels of copper and zinc (Brumbaugh et al. 1994 (**27**)). Other designs have been used to collect non-ionic organic compounds in a variety of aquatic systems (Bennett et al. 1996 (**168**); Axelman et al. 1999 (**169**)) and in overlying water (Huckins et al. 1990 (**170**)).

13.3.3.3 Peepers have also been used to expose organisms to sediments in situ (Burton et al. 2001 (**156**)). Burton et al. (1999 (**171**)) successfully introduced organisms to aerobic sediments using peepers. However, anoxic sediments are not amenable to in situ organism exposure.

13.3.3.4 Different materials might be advisable in constructing peepers depending on the contaminants of concern. For example, for many contaminants, peepers constructed from acrylic material appear to yield interstitial water samples with minimal chemical artifacts (Burton et al. 2001 (**156**)). Some polymer materials might be inappropriate for studies of certain non-ionic organic compounds. Cellulose membranes are also unsuitable, as they decompose too quickly. Plastic samplers can contaminate anoxic sediments with diffusible oxygen (Carignan et al. 1994 (**172**)).

13.3.3.5 In preparation for interstitial water collection, peeper chambers should be filled with deoxygenated water, which can be prepared by nitrogen purging for few minutes before insertion. If sediment oxidation is a concern, the peepers should be transported to the deployment site in a sealed oxygen-free water bath to minimize changes to the sediment-water equilibrium caused by dissolved oxygen interactions. However, during peeper equilibration periods, anoxic conditions are likely to be quickly reestablished. In addition, when samples are collected and processed, exposure to oxygen should be minimized. It may be useful to measure concentrations of oxygen in sediment where in situ samples are deployed for collection of interstitial water.

13.3.3.6 Following initial placement, the equilibration time for peepers may range from hours to a month, but a deployment period of one to two weeks is most often used (Adams, 1991 (**159**); Call et al. 1999 (**173**); Steward and Malley, 1999 (**174**)). Equilibration time is a function of sediment type, study objectives, contaminants of concern, and temperature (for example, Skalski and Burton, 1991(**175**); Carr et al. 1989(**176**); Howes et al. 1985(**164**); Simon et al. 1985 (**177**); Mayer, 1976 (**165**)). Membrane pore size also affects equilibration time, with larger pore sizes being used to achieve reduced equilibration times (Sarda and Burton, 1995 (**157**)). For example, using a peeper with a 149-µm pore size, Adams (1991 (**159**)) reported equilibration of conductivity within hours of peeper insertion into the sediment. Thus, it appears that equilibration time is a function of the type of contaminant, sediment type, peeper volume, and mesh pore size.

13.3.3.7 Peepers with large-pored membranes, while shortening equilibration time, also allow particulates to enter the chamber. The larger solids tend to settle to the bottom of the peeper chamber, and caution should be used to avoid collecting the solids when retrieving the water sample from the chamber. Colloidal particles will remain suspended in the sample and thereby present an artifact, but the concentration of such particles is typically lower than that found in laboratory-centrifuged samples (Chin and Gschwend, 1991 (**178**)).

13.3.3.8 In several studies, analysis of interstitial water from replicate peepers has demonstrated variable heterogeneity in water quality characteristics (Frazier et al. 1996 (**179**); Sarda and Burton, 1995 (**157**)). The potential for high variability in interstitial water chemical characteristics should be taken into account when developing the sampling design.

13.3.4 *Suction Methods*—There are a variety of suction devices for collecting interstitial water. A typical suction device consists of a syringe or tube of varying length, with one or more ports located at the desired sampling positions. The device is inserted into the sediment to the desired depth and a manual, spring-operated, or vacuum gas suction is applied to directly retrieve the water sample. A variation on this approach employs a peeper-like porous cup or perforated tube with filters. The unit is inserted into the sediment for a period of time, allowing interstitial water to infiltrate the chamber before suction is applied. The samples are then retrieved by suction. Another variation that has been used successfully employs an air stone embedded into the sediment that forces interstitial

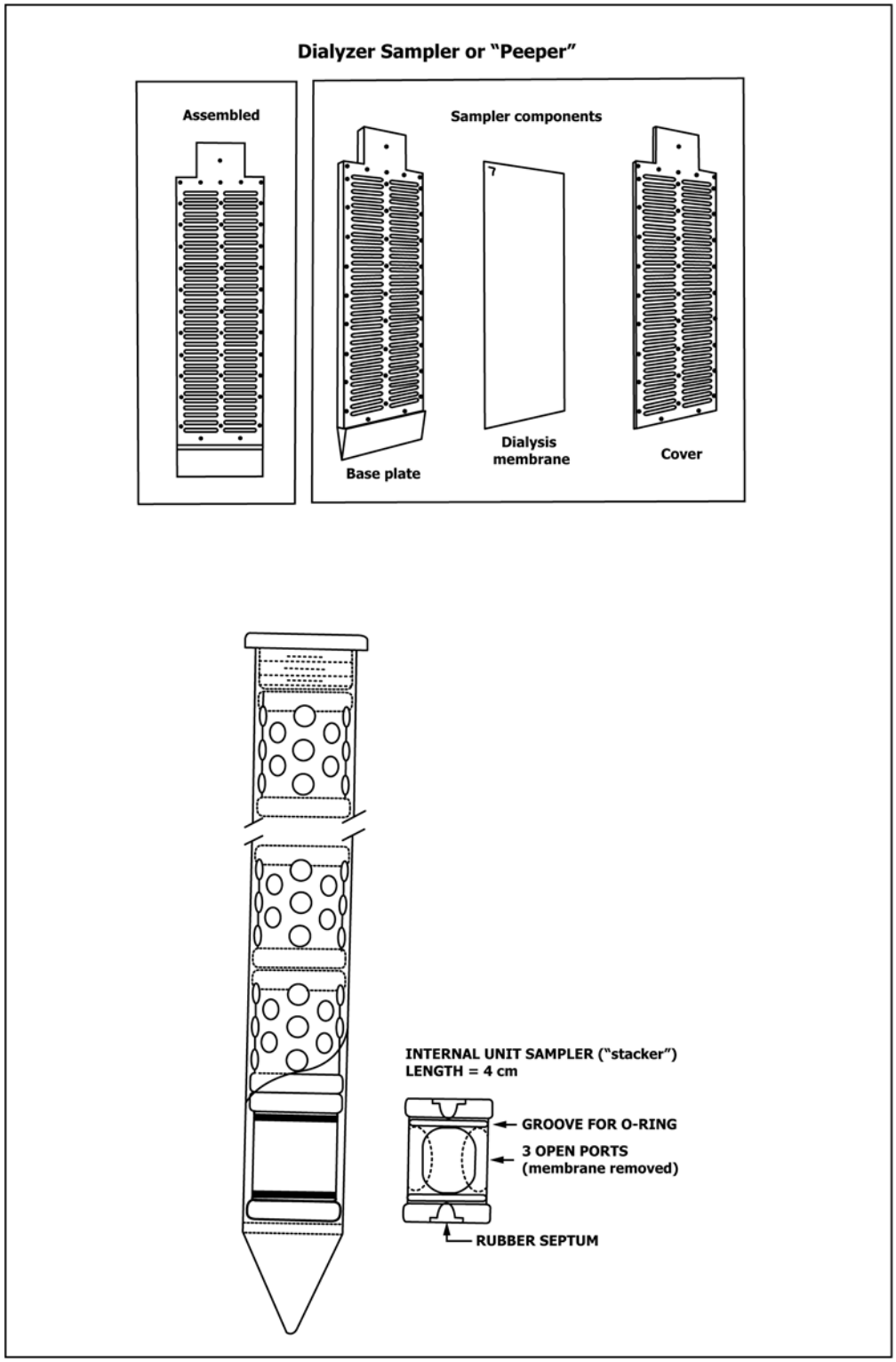


FIG. 11 Front View and Components of Peeper Sampling Devices (Top: Plate Device; Bottom: Cylindrical Probe; USEPA 2001 (1))

water upward where it can be collected via syringe or tube. All of these suction methods generally yield smaller quantities of interstitial water than peepers, and chemical (toxicological) artifacts are more likely due to greater potential exposure of interstitial water to oxygen.

13.3.5 *Processing of Field-Collected Interstitial Water Samples:*

13.3.5.1 Following sample retrieval, interstitial water might need to be recovered and stabilized quickly to prevent oxidative changes or volatilization (Carignan, 1984 (**180**)). Containers should be filled with no headspace to minimize changes in dissolved oxygen and contaminant bioavailability. Procedures for stabilization are dependent on the analyses to be performed. When non-volatile compounds are the target analytes, acidification is often stipulated, while organic carbon and methane may be stabilized with saturated mercury chloride (Mudroch and MacKnight, 1994 (**36**)). Samples for chemical analyses should be preserved immediately, if appropriate, or cooled to 4°C as soon as possible.

13.3.5.2 Samples to be analyzed for toxicity are normally cooled to 4°C as soon as possible for transport to the laboratory. USEPA methods for toxicity testing of surface waters and effluents (USEPA 1991 (**181**)) recommend that samples not be frozen in storage or transport. However, recent information suggests that freezing of interstitial water may not affect toxicity in some cases (Ho et al. 1997 (**182**), Carr and Chapman, 1995 (**183**), SETAC 2003 (**153**)). Unless a demonstration of acceptability is made for the sites of interest, interstitial water samples should not be frozen before biological testing.

13.4 *Ex-situ Extraction of Interstitial Water:*

13.4.1 Ex-situ interstitial water collection methods are often necessary when relatively large volumes of interstitial water are required (such as for toxicity testing), when in-situ collection is not viable, or when a brief sampling time is important. While these extraction methods can be done in the field or in the laboratory, extraction in the laboratory, just before analysis or testing, is preferable to maintain as close to its original state as possible during transport and storage (SETAC 2003 (**153**), Table 14). Guidance in this section reflects recommendations presented in several recent publications, including proceedings from two workshops dealing with interstitial water extraction and handling methods, and use in toxicity applications: (*1*) a dredged materials management program workshop on interstitial water extraction methods and sample storage in relation to tributyltin analysis (Hoffman, 1998 (**184**)) and (*2*) a workshop on interstitial water toxicity testing including interstitial water extraction methods and applications (SETAC 2003 (**153**)).

TABLE 14 Recommended Procedures for Extraction of Interstitial Water in the Laboratory (USEPA 2001 (1))

Centrifugation is the generally preferred laboratory method for the extraction of interstitial water.
Extraction of interstitial water should be completed as soon as possible.
Interstitial water that has accumulated on the surface of the homogenized sediment sample should be mixed into the sediment before the sample is partitioned among centrifuge bottles.
Unless other program-specific guidance is available, sediments should be centrifuged at high speed (for example, 8000 to 10 000 × g) for 30 min.
Unless site-specific information suggests otherwise, centrifuging should be at 4°C to minimize temperature-mediated biological and chemical processes.
Interstitial water should be preserved immediately for chemical analyses or analyzed as soon as possible after extraction, unpreserved. For toxicity testing, interstitial water should be stored at 4°C for not longer than 24 h, unless the test method dictates otherwise.
Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead.

13.4.2 *General Procedures:*

13.4.2.1 Centrifugation and squeezing are the two most common techniques for collecting interstitial water, and are generally preferred when large volumes are required. Other methods include pressurization (for example, sediment squeezing, 13.3.4 or vacuum filtration, 13.3.5) devices, which can be used to recover small volumes of interstitial water.

13.4.2.2 Regardless of the method used, interstitial water should be preserved immediately for chemical analyses, if appropriate, or analyzed as soon as possible after sample collection if unpreserved (such as for toxicity testing; Hoffman, 1998 (**184**); SETAC 2003 (**153**)). Significant chemical changes can occur even when interstitial water is stored for periods as short as 24 h (Hulbert and Brindle, 1975 (**185**); Watson et al. 1985 (**186**); Kemble et al. 1999 (**89**); Sarda and Burton, 1995 (**157**); SETAC 2003 (**153**)).

13.4.2.3 If sediments are anoxic, as most depositional sediments are, sample processing, including mixing of interstitial water that has separated from the sediment, should be conducted in an inert atmosphere or with minimal atmospheric contact. Exposure to air can result in oxidation of contaminants, thereby altering bioavailability (Bray et al. 1973 (**187**); Lyons et al. 1979 (**188**); Howes et al. 1985 (**164**)). Air exposure can also result in loss of volatile sulfides, which might increase the availability of sulfide-bound metals (Allen et al. 1993 (**189**); Bufflap and Allen, 1995 (**158**)). In addition, iron and manganese oxyhydroxides are quickly formed upon exposure to air. These compounds readily complex with trace metals, thus altering metals-related toxicity (Bray et al. 1973 (**187**); Troup et al. 1974 (**190**); Burton, 1991 (**129**); Bufflap and Allen, 1995 (**158**)). Maintaining anoxic processing conditions is not necessary when study objectives are concerned with exposures to aerobic sediments, or if target contaminants are unaffected by oxidation in short-term toxicity testing.

13.4.3 *Centrifugation:*

13.4.3.1 Centrifugation is the generally preferred laboratory method for collection of interstitial water (SETAC 2003 (**153**)). It is a relatively simple procedure that allows rapid collection of large volumes of interstitial water. It also facilitates the maintenance of anoxic conditions (if required). However, centrifugation, like other ex-situ procedures, might yield chemical or toxicological artifacts due to the extraction procedures themselves, which might alter the natural equilibrium between interstitial water and sediment.

13.4.3.2 Before centrifugation, the sediment sample is homogenized and placed into centrifuge bottles. If the homogenized sample is stored before centrifugation, interstitial water might accumulate on the surface of the sediment. This overlying water should be mixed into the sediment before subsampling for centrifugation. Samples are then partitioned among centrifuge bottles. In general, about 50 % of sediment moisture

content can be extracted as interstitial water. If interstitial water volume requirements are lower, smaller sediment subsamples can be used.

13.4.3.3 Interstitial water has been isolated over a range of centrifugal forces and durations (Landrum et al. 1987 (**191**); Giesy et al. 1988 (**192**); Schults et al. 1992 (**139**); Burgess et al. 1993 (**47**); Ankley et al. 1990 (**193**); Schubauer-Berigan and Ankley,1991 (**194**); Ankley and Schubauer-Berigan, 1994 (**195**); Kemble et al 1994 (**73**)). For toxicity testing of interstitial waters, some sources recommend that sediments be centrifuged at 10 000 × g for a 30 min period (Environment Canada, 1994 (**2**)). Such high speed centrifugation is often necessary to remove most colloids and dispersible clays (Adams, 1991 (**159**) ; Chin and Gschwend, 1991 (**178**) ; Brownawell and Farrington, 1986 (**196**); Ankley and Schubauer-Berigan, 1994 (**195**)), which can introduce interferences to chemical or toxicological analysis. However, such high speed centrifuges are not commonly available. Furthermore, many materials (glass, plastic) are not able to withstand high centrifugation speeds. Finally, it should be noted that toxicity is typically reduced with high speed centrifugation due to the removal of particle-associated contaminants (Sasson-Brickson and Burton 1991(**197**); Schults et al. 1992 (**139**); Ankley and Schubauer-Berigan, 1994 (**195**) ; Bufflap and Allen, 1995 (**158**)).

13.4.3.4 Based on research to date, both slower and faster centrifugation speeds (and associated differences in colloid or suspended solids removal) may be appropriate depending on the study objectives. High speed centrifugation may not be appropriate because one is interested in toxicity potential of the interstitial water in its entirety (that is, including colloidal material). However, if one is interested in comparing interstitial water contaminant concentrations to model exposure compartments for example (EPRI, 2000 (**198**)), then high speed centrifugation might be necessary. As our knowledge is still limited in this area, it is perhaps most important to note that centrifugation speed can have an effect on the observed toxicity and chemical characteristics. Therefore, a consistent centrifugation procedure (including speed and time) should be identified and used throughout a study for all samples.

13.4.3.5 Centrifugation has been performed at various temperatures. It may be desirable to select a centrifugation temperature that reflects the in situ sediment temperature so that equilibrium between the particulate and interstitial water is not substantially altered. Alternatively, a temperature of 4°C may be preferred to minimize temperature-mediated chemical and biological processes (Environment Canada, 1994 (**2**)).

13.4.3.6 When centrifuging coarse sand, it might be desirable to use a modified centrifuge bottle to aid interstitial water recovery (USEPA/USACE, 1998 (**35**)). The modified bottle is equipped with an internal filter that can recover 75 % of the interstitial water, as compared to 25 to 30 % recovery from squeezing (Saager et al. 1990 (**199**)).

13.4.3.7 As discussed in 11.2, all containers have limitations with regards to adsorption or leaching of chemicals, ease of use, and reliability. For example, polytetrafluororthylene (PTF) bottles have been used successfully up to 2500 × g when filled to 80 % of capacity, but collapse at 3000 g (Burgess et al. 1993 (**47**)). Polycarbonate bottles have been used successfully for tributyltin analyses in interstitial water (Hoffman, 1998 (**184**)). If small volumes of water are required for testing, higher speed centrifugation can be performed with glass tubes (up to 10 000 g, Word et al. 1987 (**105**)). Larger glass tubes, however, can not be centrifuged at such high speeds. If metal toxicity is not a concern, then high speed centrifugation in larger stainless steel centrifuge tubes is suitable. If test samples are contaminated with photoreactive compounds such as PAHs, exposure of the sample to light should be minimized to limit degradation or alteration of potentially toxic compounds.

13.4.4 *Sediment Squeezing:*

13.4.4.1 Isolation of interstitial water by squeezing has been performed using a variety of procedures and devices (Reeburgh, 1967 (**200**); Kalil and Goldhaker, 1973 (**201**); Jahnke, 1988 (**202**); Carr et al. 1989 (**176**); Long et al. 1990 (**28**); Watson and Frickers, 1990 (**163**); Adams, 1991 (**159**); Carr and Chapman, 1995 (**183**); Carr, 1998 (**155**)). Low-pressure mechanical squeezers can be constructed, and may provide specialized capacities such as collection of interstitial water profiles from core samples (Bender, et al. 1987(**203**)). In all cases, the interstitial water is passed through a filter that is a part of the squeezing apparatus.

13.4.4.2 Squeezing has been shown to produce a number of artifacts due to shifts in equilibrium from pressure, temperature, and gradient changes (for example, Froelich et al. 1979 (**204**); Kriukov and Manheim, 1982 (**205**); Bollinger et al. 1992 (**206**); Schults, 1992 (**139**)). Squeezing can affect the electrolyte concentration in the interstitial water particularly with a decrease in chemical concentrations near the end of the squeezing process. However, others reported that squeezing did not produce artifacts in interstitial water toxicity studies (Carr and Chapman 1995 (**183**); Carr 1998 (**155**); SETAC 2003 (**153**)). It is therefore recommended that if squeezing is performed, moderate pressures be applied along with electrolyte (conductivity) monitoring during extraction (Kriukov and Manheim, 1982 (**205**)). Squeezing should also be performed at in situ ambient temperatures, as significant alterations to interstitial water composition can occur when squeezing is conducted at temperatures different from ambient conditions (for example, Mangelsdorf et al. 1969 (**207**); Bischoff et al. 1970; Sayles et al. 1973 (**208**)).

13.4.4.3 Other sources of interstitial water alteration during squeezing are: contamination from overlying water; internal mixing of interstitial water during extrusion; and solid-solution reactions as interstitial water is expressed through the overlying sediment. As interstitial waters are displaced into upper sediment zones, they come in contact with solids with which they are not in equilibrium. This inter-mixing causes solid-solution reactions to occur. Most interstitial water chemical species are rapidly transformed, as observed with ammonia and trace metals (Rosenfield, 1979 (**209**); Santschi et al. 1997 (**210**)). Bollinger et al. (1992) (**206**) found elevated levels of several ions and dissolved organic carbon in squeezed samples as compared to samples collected by in situ peepers. The magnitude of the artifact will depend on the characteristics of the contaminant and redox potential.

13.4.5 *Pressurized and Vacuum Devices:*

13.4.5.1 Other methods for extraction of interstitial water from sediment samples can include vacuum filtration (Jenne and Zachara, 1987 (**130**); Knezovich and Harrison, 1987 (**211**); Winger and Lasier, 1991 (**212**)), gas pressurization (Reeburgh, 1967 (**200**)), and displacement (Adams, 1991 (**159**)). These methods typically recover only small volumes of interstitial water and are not commonly used.

13.4.5.2 Use of a hand vacuum with an aquarium stone is an effective vacuum filtration method (Winger and Lasier, 1991(**212**); Sarda and Burton, 1995 (**157**)). The procedure typically involves attaching the air stone to a 50 mL syringe via plastic tubing, inserting it into the sediment to the desired depth, and then applying suction. This method can recover relatively large volumes of interstitial water; Santschi et al. (1997 (**210**)) used this procedure to extract up to 1,500 mL from 4 L of sediment. Sarda and Burton (1995 (**157**)) found that ammonia concentrations in water obtained by this procedure were similar to those collected by in situ peepers. Drawbacks to this method include loss of equilibrium between the interstitial water and the solids, filter clogging, and oxidation (Brinkman et al. 1982 (**213**)).

14. Physicochemical Characterization of Sediment Samples

14.1 *General Information*—It is often necessary or desirable to determine certain physicochemical characteristics of sediments in the laboratory, in conjunction with toxicity testing or chemical analysis for inorganic or organic contaminants. This characterization should include measurement of certain parameters known to mediate the availability of contaminants in sediment (Test Method E1706). Bulk chemical concentrations alone should not be used to evaluate bioavailability (USEPA 1998 (**214**)). The following parameters are generally measured: pH (pore water), ammonia (pore water), total organic carbon, particle size distribution (for example, percent sand, silt and clay), percent water content, salinity or hardness of pore water, and conductivity of pore water. Depending on the experimental design or study objectives, more extensive characterization may be necessary. Several additional characteristics that may assist in study implementation, data interpretation, or QA/QC (that is, assessing sediment integrity, artifact production, optimal extraction and test procedures) include: sediment biochemical oxygen demand (BOD), sediment chemical oxygen demand (COD), sediment oxygen demand (SOD), cation exchange capacity (CEC), Redox (Eh) or oxidation-reduction potential (ORP), total inorganic carbon, total volatile solids, acid volatile sulfides (AVS), simultaneously extracted metals (SEM), metals, petroleum hydrocarbons, other organic compounds (pesticides, PCBs, PAHs, and TCDD-dioxin), oil and grease, and dissolved organic carbon (DOC) in the pore water. Measurements of many sediment physicochemical characteristics use analytical techniques originally developed for soils and waters, and the literature should be consulted for details regarding recommended methodology (Black, 1965 (**215**); USGS, 1969 (**216**); Plumb, 1981(**49**); Page et al. 1982 (**217**)). The following sections provide rationale for making each type of sediment physicochemical measurement, along with brief descriptions of measurement techniques, and references for further information and specific procedures.

14.2 *pH in Pore Water:*

14.2.1 Sediment pH is often one of the single most important factors controlling speciation and equilibria for many chemicals including sulfides, ammonia, cyanide, and metals, all of which ionize under the influence of pH. The USEPA ammonia water-quality criterion, for example, is dependent in part on pH because ammonia toxicity is largely governed by the unionized ammonia fraction which is pH-dependent (USEPA, 1999 (**218**)). Metal (Cd, Cu, Ni, Pb, and Zn) speciation and bioavailability are also known to be affected by pH (Schubauer-Berigan and Ankley, et al. 1991 (**194**); Ho et al. 1999 (**219**)).

14.2.2 Generally, pH is measured using a pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature compensating device. A circuit is completed through the potentiometer when the electrodes are submersed. General purpose process pH electrodes are available in a wide variety of configurations for in-line and submersion applications. Generally, electrodes with gel-filled references require less maintenance than electrodes with liquid-filled references. The latest instruments have microprocessors that automatically calculate and display the slope. Some older instruments have a percent-slope readout or (and) millivolt readout. For instruments with a millivolt readout, the measured electrode potential is calculated as the difference between millivolts measured at the known pH of two buffers.

14.2.3 Plumb (1981 (**49**)) and Gonzalez (1995 (**220**)) described a method for measuring pH in sediment using a pH probe and meter. The probe was inserted into the sediment and pH directly measured after at least a 5 min equilibration time. Electrodes have also been used for direct measurements of pH in sediment pore water, or in a 1 to 1 mixture of sediment to water (Jackson, 1958 (**221**)). Direct measurement of sediment pH is also possible using electrodes with “spear tip” designs allowing for greater penetration into the sample. Detailed methods for measuring pH in water and sediment are also described by USEPA (1979 (**222**);1983 (**55**);1986 (**223**);1987 (**43**)).

14.3 *Ammonia in Pore Water:*

14.3.1 Nitrogen, a nutrient associated with over-enrichment of aquatic environments, exists in several forms, including ammonia. Ammonia is highly soluble in water where it is found in an un-ionized form (NH_3) and in an ionized form as NH_4+. The extent of ionization is dependent on pH, temperature, and salinity (in seawater). Ammonia in sediments and pore water is generally the result of microbial degradation of nitrogenous organic material such as amino acids (Ankley et al. 1990 (**193**)). Pore water concentrations of ammonia as high as 50 mg/L have been measured in otherwise uncontaminated sediments (Murray et al. 1978 (**224**); Kristensen and Blackburn, 1987 (**225**)), while ammonia in pore waters from contaminated sediments can range from 50 to more than 200 mg/L (Ankley et al. 1990 (**193**); Schubauer-Berigan and Ankley, 1991 (**194**)). Elevated concentrations of ammonia (Sims et al. 1995a (**226**); Moore et al. 1997 (**227**)) and hydrogen sulfide (Sims et al. 1995b (**228**)) have frequently been found in deeper dredged sediment samples compared to surficial sediment samples.

14.3.2 The toxic effects of ammonia are generally considered to be associated with the un-ionized fraction (NH3) rather than the ionic components (NH_4+ and NH_4SO_4-), which co-exist in equilibria. This equilibrium is highly dependent on pH, temperature, pressure, salinity, and ionic concentrations of ammonia. The more toxic un-ionized ammonia fraction can be calculated using known total ammonia values and measurements of pH, pressure, salinity, and temperature as described by Whitfield (1978 (**229**)) and Thurston et al (1981 (**230**)).

14.3.3 USEPA (1983 (**55**)), and APHA (1995 (**231**)) describe five methods available to measure ammonia in the pore water: the titrimetric method; the ammonia-selective electrode method; the ammonia-selective electrode method using known addition; the phenate method; and, the automated phenate method.

14.3.4 A preliminary distillation step may be required if interferences are present (APHA, 1995 (**231**)). Interferences (for example, sample constituents that interact with procedural reagents) are described in detail in the APHA 1995 (**231**) and Guide D1426. Once distilled, the sample can be analyzed using any of the methods listed above.

14.3.5 The distillation and titration methods are frequently used when ammonia concentrations are greater than 5.0 mg/L. The ammonia-selective electrode method is appropriate when concentrations range between 0.03 and 1400 mg NH_3-N/L. Ammonia readings are calibrated against ammonia standards. To verify meter readings, confirmatory subsamples can be preserved and analyzed for ammonia using the standard Nessler technique described in APHA (1995) (**231**). For the phenate method, APHA (1995) (**231**) recommends distillation with sulfuric acid when interferences are present (Bower and Holm-Hansen, 1980 (**232**)). The automated phenate method is suitable for pore waters with ammonia concentrations in the range of 0.02 and 2.0 mg NH_3-N/L.

14.4 *Total Organic Carbon Content (TOC):*

14.4.1 The total organic carbon (TOC) content of sediment is a measure of the total amount of oxidizable organic material. TOC is the sum of dissolved organic carbon (DOC), particulate organic carbon (POC) or suspended organic carbon (SOC), and colloids. TOC is an important parameter to measure in sediments because it is a major determinant of non-ionic organic chemical bioavailability (Di Toro et al. 1991 (**90**)). Metal bioavailability is also affected by the amount of TOC present in sediments. TOC is usually expressed as a percentage of the bulk sediment, and is used to normalize the dry-weight sediment concentration of a chemical to the organic carbon content of the sediment. USEPA Equilibrium Partitioning Guidelines estimate bioavailability as a function of contaminant concentration sorbed to sediment organic carbon and contaminant concentration in the pore water under equilibrium conditions (USEPA, 1998 (**214**)). Recently, the presence of soot carbon from the combustion of organic carbon (for example, fossil fuels) has been recognized as a fraction of the TOC in sediment. Soot carbon may alter the geochemistry and bioavailability of some organic contaminants (Gustuffson et al. 1997 (**233**)). Methods for determining organic carbon in sediment have been reviewed (Schumacher 2002 (**234**)).

14.4.2 The organic carbon content of sediments has been measured using several methods including: wet oxidation titration, modified titration, and combustion after removal of carbonate by the addition of HCl and subsequent drying. USEPA methods (1986 (**223**); 1987 (**43**)), including SW-846 and 430/9-86-004, are often used to measure TOC. Plumb (1981) (**49**) recommends one of two methods to separate organic from inorganic carbon before analyzing for TOC: (*a*) ignition and using HCl as the acid for pre-treating sediment, or (*b*) differential combustion, which uses thermal combustion to separate the two forms of carbon.

14.4.3 USEPA/USACE guidance (1998) (**35**) recommends that TOC analyses be based on high-temperature combustion rather than on chemical oxidation because some classes of organic compounds are not fully degraded by combined chemical and ultraviolet oxidation techniques. Inorganic carbon (for example, carbonates and bicarbonates) can be a significant proportion of the total carbon in some sediments. Therefore, samples should be treated with acid to remove the inorganic carbon before TOC analysis. The procedure described by the Puget Sound Estuary Program (PSEP, 1997a(**34**)) is recommended for TOC analysis because this method uses high-temperature combustion using an induction furnace. USEPA recommends a similar method using catalytic combustion and non-dispersive infrared detection (Leonard, 1991 (**235**)) for quantifying TOC. Because of interferences associated with TOC measurement in high carbonate sand areas in Florida and in Hawaii, some investigators have not been able to use acid addition to remove inorganic carbon and have instead used the Lloyd Kahn method (Kahn 1988 (**236**); David Moore MEC Analytical, Carlsbad, CA; personal communication).

14.4.4 Several methods for measuring the total organic carbon (TOC) content of sediments exist (See Nelson and Sommers 1996 (**237**) for a review). However, acceptable methods should at a minimum include the following steps:

14.4.4.1 *Sample Collection*—Sediment samples are collected and stored in non-organic containers.

14.4.4.2 *Sample Preparation*—Each sediment sample should have macroscopic pieces of shells (for example, >1 mm) removed and then be pulverized and homogenized. Each sediment sample should be treated by direct addition with a strong non-oxidizing acid (for example, HCl) for about 18 h to remove inorganic carbon; sample pH should be about 2 after acidification (Yamamuro and Kayanne, 1995 (**238**)). Each sediment sample is oven dried following acid treatment (60 to 70°C; Weliky et al. 1983 (**239**); Yamamuro and Kayanne, 1995 (**238**)). Each sediment sample is stored in a desiccator until analysis. As noted, desiccation is highly recommended, however if not possible, a pre- and post-acidification sample weight should be performed to correct for water uptake (Hedges and Stern, 1984 (**240**)).

14.4.4.3 *Sample Analysis*—Each post-acidification sediment sample should be analyzed using acceptable instrumentation. Instrumentation should have a detection limit of about 100 mg/Kg. Quantification of organic carbon should be based on a sample's weight, measured before acidification.

14.5 *Particle Size Distribution (Percent Sand, Silt, and Clay):*

14.5.1 Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological characteristics, it can be used to normalize chemical concentrations and account for some of the variability found in biological assemblages (USEPA 1998 (**214**)) or in laboratory toxicity testing (USEPA, 2000d (**30**); Hoss et al. 1999 (**241**)). Particle size can be characterized in varying detail. The broadest divisions that generally are considered useful for characterizing particle size distributions are percentages of gravel, sand, silt, and clay. However, each of these size fractions can be subdivided further so that additional characteristics of the size distribution are determined (PSEP, 1996 (**242**)).

14.5.2 Particle size determinations can either include or exclude organic material. If organic material is removed before analysis, the "true" (that is, primarily inorganic) particle size distribution is determined. If organic material is included in the analysis, the "apparent" (that is, organic plus inorganic) particle size distribution is determined. Because true and apparent distributions may differ, detailed comparisons between samples analyzed by these different methods are questionable. Therefore, if comparisons among samples between studies is desired, sediment particle size should be measured using consistent methods (PSEP, 1996 (**242**)). For interpretation of biological effects or chemical partitioning in sediment, the "apparent" particle size distribution may be more desirable to determine compared to the measurement of the "true" particle size distribution (Word et al. 2004 (**242**)).

14.5.3 Sediment particle size can be measured by a number of different methods (Allen, 1975 (**243**); Plumb,1981 (**49**) ; PSEP, 1996 (**242**)). The best method will depend on the particle properties of the sample (Singer et al. 1988 (**244**)). Particle size distribution is often determined by either wet sieving the sample (USEPA, 1979 (**222**); Plumb, 1981 (**49**) ; PSEP, 1996 (**242**); Singer et al. 1988 (**244**)), the hydrometer method (Day, 1965 (**245**); Patrick, 1958 (**246**)), the pipette method (USGS, 1969 (**216**); Rukavina and Duncan, 1970 (**247**)), settling techniques (Sanford and Swift, 1971 (**248**)), by use of laser diffraction, or X-ray absorption (Duncan and Lattaie, 1979 (**249**); Rukavina and Duncan, 1970 (**247**)). The pipet method may be superior to the hydrometer method (Sternberg and Creager, 1961 (**250**)). Combinations of multiple methods may provide refined measurements of particle size distribution. Gee and Bauder (1986 (**251**)) used sieving and pipeting after soluble salts were removed. Gonzalez (1995 (**220**)) used a combination of sieve and hydrometer methods. Folk (1968 (**252**)_ and Buchanan (1984 (**253**)) discuss additional methods to measure particle size.

14.5.4 Recommended methods for measuring sediment particle size distribution are those of PSEP (1996 (**242**)) and USEPA (1995 (**254**). Percent gravel, sand, silt, and clay are determined as apparent distribution using a minimum sediment sample size of 100 g taken from a homogenized sediment sample. Organic matter should be removed before analysis by oxidation using hydrogen peroxide. Wet-sieving followed by dry sieving (mechanical shaking) separates the two coarse particle size groups. The silt-clay fraction is subdivided using a pipet technique that depends upon the differential settling rates of the two different particle size fractions. All fractions are dried to a constant weight. Cooled samples are stored in a desiccator and weighed.

14.5.5 Particle analyzers may be preferable over pipette methods for the evaluation of fine fractions due to the introduction of human error in the sampling process (for example, Beckman Coulter LS100Q laser diffraction particle size analyzer or Micromeritics Sedigraph; Syvitski et al. 1991(**255**)). To obtain an accurate determination of particle sizes for the fine fraction, the Coulter (particle size) counter method may be employed (McCave and Jarvis, 1973 (**256**); Vanderpleog, 1981 (**257**)). This method gives the fraction of particles with an apparent spherical diameter. In a review of the available methods, Swift et al. (1972) (**258**) found the Coulter counter method to be the most versatile method overall; however, it does not provide settling information. Another potential method for determining the particle size distribution of a very fine fraction is through the use of electron microscopy (Leppard et al. 1988 (**259**)). Collection techniques for very fine material can result in aggregation of larger colloidal structures (Leppard, 1986 (**260**); Leppard et al. 1988 (**259**)). In general, particle settling methods are preferred to sediment sizing methods. Unless there is a large amount of organic matter, particle size should be determined with the organic matter present.

14.6 *Percent Water or Moisture Content:*

14.6.1 Water content is a measurement of sediment moisture usually expressed as a percentage of the whole sediment weight. Sediment moisture content is measured as the difference between wet weight of the sediment and dry weight following oven drying at 50 to 105°C to a constant weight. Percent water is used to convert sediment concentrations of substances from wet-weight to a dry-weight. Methods for determining moisture content are described by Plumb (1981 (**49**)) and Vecchi (1999 (**261**)). Additional methods are provided in USEPA (1987 (**43**)).

14.7 *Salinity of the Pore Water (Marine Sediments):*

14.7.1 Salinity is a measure of the mass of dissolved salt in a given mass of solution. The most reliable method to determine the true or absolute salinity is by complete chemical analysis. However, this is time consuming and costly. Therefore, indirect methods are more suitable. Indirect methods include conductivity, density, sound speed, or refractive index (APHA, 1995) (**231**). Salinity is then calculated from the empirical relationship between salinity and the indirect measurement. Conductivity measurements have the greatest precision, but respond only to ionic solutes (APHA, 1995 (**231**)). Density measurements respond to all solutes. APHA (1995 (**231**)) recommends the electrical conductivity method, because it is sensitive and easily performed. APHA (1995 (**231**)) also recommends the density method, using a vibrating flow densitometer.

14.7.2 A salinity refractometer can be used for quick readings of salt density in solutions such as sea water. These refractometers are easy to read, non-corrosive, and lightweight. They have dual scales and an adjustable focus. Temperature

and non-temperature compensating refractometers are available. Most refractometers are accurate to 1 ppt and read specific gravity (1.000 to 1.070 in 0.001 divisions) and parts per thousand (0 to 100 in 1 part per thousand divisions).

14.8 *Conductivity of the Pore Water (Freshwater Sediments):*

14.8.1 Conductivity is a measure of the ability of an aqueous solution to carry an electric current. This ability is dependent on the presence of ions in the solution, the concentration of the ions, their mobility and valence, and temperature. Solutions of inorganic compounds are usually good conductors while those of organic compounds are usually poor conductors. Conductivity is enhanced by calcium, potassium, sodium, and magnesium chlorides and sulfides. Meters can be used to measure the degree to which electrical current can travel through water. The unit of measure is 1 mS/m = 1 millisiemens/metre or 1 µS/cm = 1 microsiemens/centimetre. The reading indicates the amount of ions in the water. While traditional chemical tests for hardness measure calcium and magnesium, they fail to provide an indication of other ions (for example, sodium). The conductivity meter provides a much better measure of ionic strength.

14.9 *Acid Volatile Sulfide (AVS):*

14.9.1 Measurement of acid volatile sulfides (AVS) and simultaneously extracted divalent metal (SEM) concentrations associated with AVS extraction can provide insight into the bioavailability of metals in anaerobic (anoxic) sediments (Di Toro et al. 1990 (**53**); Ankley et al. 1996 (**54**)). AVS is the reactive solid-phase sulfide fraction that is extracted by cold hydrochloric acid. AVS appears to affect the bioavailability of most divalent metal ions as the sulfide ions have a high affinity for divalent metals. This affinity results in the formation of insoluble metal sulfides with greatly reduced bioavailability. AVS concentrations in freshwater and marine sediments can range from less than 0.1 to greater than 50 µmol AVS/g of sediment (Di Toro et al. 1990 (**53**)).

14.9.2 The bioavailability of metals in sediments has been predicted by comparing the molar concentration of AVS to the molar concentration of SEM (methods described below). If AVS is greater than SEM, the metals are bound in sulfide complexes with greatly limited bioavailability. However, if SEM is greater than AVS, metals may or may not be toxic due to other controlling factors (for example, TOC).

14.9.3 The easily extractable sulfide fraction can be measured using the acid purge and trap technique. The sample sulfide is solubilized in cold hydrochloric acid. The analytical method involves conversion of sulfides to aqueous H_2S. This may be measured with a sulfide probe or by following a wet chemistry method. In the latter method, silver sulfide is precipitated in a gas-tight assembly and flushed with nitrogen to eliminate oxidation. The precipitate is filtered, dried, and weighed. The weight is compared with the weight obtained from a non-acidified sample, and the difference is attributed to the AVS fraction (Di Toro et al. 1990 (**53**)).

14.10 *Simultaneously Extracted Metals:*

14.10.1 A model for predicting toxicity from divalent trace metals (Di Toro et al. 1990 (**53**)) is based on the binding of these metals to AVS. Where the sum of the moles of the SEM, including Ag, Cd, Cu, Ni, Pb, and Zn is exceeded by the molar concentration of AVS, the metals are insoluble and largely unavailable to biota. The extraction of AVS and SEM metals should be achieved using a single methodology so that recoveries associated with each measure are consistent. Simultaneous extraction improves the efficiency of the methodology.

14.10.2 SEM can be measured in filtered aliquots by atomic absorption methods (Di Toro et al. 1990 (**53**)). Recent SEM analysis methods use inductively coupled plasma atomic emission spectrometry (ICP-AES; Berry et al. 1999 (**262**)). Other methods for analysis of metals are described in 14.11.

14.11 *Metals:*

14.11.1 Low levels of trace metals occur naturally in the environment but highly elevated levels in sediment are generally associated with anthropogenic contaminant loads. Metals are partitioned in sediments as soluble free ions, soluble organic and inorganic complexes, easily exchangeable ions, precipitates of metal hydroxides, precipitates with colloidal ferric and manganic oxyhydroxides, insoluble organic complexes, insoluble sulfides, and residual forms (Gambrell et al. 1976 (**263**)).

14.11.2 Current instrument methods available for the analysis of trace metals include electrochemistry (for example, differential pulse polarography), spectrophotometry (for example, silver diethyldithiocarbamate), atomic absorption spectrophotometry, atomic emission spectrophotometry, x-ray fluorescence (XRF), and neutron activation (PSEP 1997b (**264**)). The most commonly used instrumental method to analyze sediments for metals is atomic absorption spectrophotometry (PSEP, 1997b (**264**)). Inductively coupled plasma mass spectrometry (ICP-MS) or ICP-AES allow for simultaneous determination of many metals at concentrations below a part per billion with little pretreatment (Crecelius et al. 1987 (**265**); Berry et al. 1999 (**262**)).

14.11.3 The concentration of salt in marine or estuarine samples may interfere with metals analyses (USEPA/USACE, 1998(**35**)). Therefore, acid digestion and atomic absorption spectroscopy should be coupled with an appropriate technique to control for this interference. Methods in USEPA (1986 (**223**)) are recommended for the analysis of mercury in sediments and EPRI (1986 (**266**)) methods are recommended for the analysis of selenium and arsenic. USEPA methods for cadmium, hexavalent chromium, copper, lead, mercury, nickel, selenium, silver, and zinc are described by USEPA (1986 (**223**)). PSEP (1997b (**264**)) suggests that mercury can be extracted using vacuum distillation and analyzed by gas chromatography/mass spectrophotometry.

14.12 *Petroleum Hydrocarbons and Polycyclic Aromatic Hydrocarbons:*

14.12.1 Petroleum hydrocarbons are oil and grease constituents which remain in solution after contact with silica gel. Petroleum distillates, also called hydrocarbons or petrochemicals, refer to a broad range of compounds that are extracted by distillation during the refining of crude oil. During the fractional distillation of petroleum, crude oil is heated to allow various compounds to turn from liquid into gas, and then are captured as they rise, cool, and condense. Lighter, more

volatile compounds rise higher before they condense and are collected on distillation trays. Heavier, less volatile compounds such as diesel fuel and oil are collected on lower distillation trays. Waxes and asphalts are collected from the bottom after the other products have volatilized.

14.12.2 Petroleum distillates contain both aromatic hydrocarbons (carbon rings) and aliphatic hydrocarbons (straight carbon chains). The chemical structure of the hydrocarbon largely defines the nature and behavior of these compounds. Aromatic hydrocarbons are the most toxic compounds found in petroleum products. Most aromatic hydrocarbons are chronic toxicants and known carcinogens. Aromatic compounds are found in all crude oils and most petroleum products. Many aromatic hydrocarbons have a pleasant odor and include such substances as naphthalene, xylene, toluene, and benzene. Aliphatic hydrocarbons are flammable and may be explosively flammable. Aliphatic hydrocarbons include methane, propane, and kerosene.

14.12.3 Aromatic and aliphatic hydrocarbons were analyzed in sediments by Page et al. (1995a, b (**44**),(**45**)). Sediment samples were spiked with the appropriate surrogates, mixed with equal amounts of sodium sulfate to dry the samples, and extracted with a methylene chloride acetone mixture (Method 3550, USEPA, 1986 (**223**)). The concentrated extracts were partitioned on an alumina column into saturated and unsaturated hydrocarbon fractions (Method 3611, USEPA, 1986 (**223**)). The fractions were concentrated using the appropriate pre-injection volume, spiked with the appropriate internal standards, and analyzed by gas chromatography with flame ionization detection (GC/FID) and gas chromatography with mass spectrometry detection (GC/MS) operating in the selected ion monitoring (SIM) mode. The method of internal standards (Method 8000, USEPA, 1986 (**223**)) using the average relative response factors generated from the linear initial calibration was used to quantify the target compounds. All data were corrected for the recovery of the appropriate surrogate compound. Their relative abundances could then be used for identification and quantification purposes.

14.12.4 TPH (total petroleum hydrocarbons) and PAH (polycyclic aromatic hydrocarbons) have also been analyzed by first acidifying the sample with concentrated hydrochloric acid and then extracting hydrocarbons with a mixture of methanol and hexane. The hexane extracts were then spiked with an internal standard and analyzed by GC-FID for TPH content and by GC/mass spectrometry (MS) for PAH analysis.

14.12.5 Kaplan et al. (1996 (**267**)) extracted hydrocarbons using anhydrous Na_2SO_4 with methylene chloride and sonication. The total solvent extract was then concentrated with Kuderna-Danish equipment. The concentrate was further concentrated using a gentle stream of dry nitrogen. An aliquot was then injected directly into the gas chromatography.

14.13 *Other Organic Compounds (Pesticides, PCBs, TCDD-Dioxin):*

14.13.1 Analytical techniques for measuring organic compounds require five general steps: drying the sample, extraction, drying the extract, clean up of the extract, and analysis of the extract. PSEP (1997c (**268**)) recommends centrifugation or sodium sulfate to dry the sample and a solvent extraction, with application of shaker/roller, or sonication. Sample drying with sodium sulfate is recommended for samples weighing about 10 g (after overlying water is decanted). The sediment and sulfate mixture is extracted and the extract is processed (MacLeod et al. 1985 (**269**)).

14.13.2 Soxhlet® extraction (USEPA, 1986 (**223**)) involves distillation with a solvent such as acetone, dichloromethane/methanol (2:1), dichloromethane/methanol (9:1), and benzene/methanol (3:2). USEPA (1983 (**55**)) recommends sonication with solvent mixtures and a 30-g subsample of sediment.

14.13.3 Drying the extract can be accomplished through separatory funnel partitioning as needed to remove water and sodium sulfate or by using a Kuderna-Danish apparatus and rotary evaporation with purified nitrogen gas for concentration to smaller volumes (PSEP, 1997c (**268**)). Using the separatory funnel partitioning method, the wet sample is mixed with methanol and centrifuged. The supernatant is decanted and extracted later. Extraction of the sample is continued using less polar solvents and the water/methanol and solvent extracts are combined and dried.

14.13.4 According to PSEP (1997c (**268**)) elemental sulfur can be removed from the sediment sample with vigorous mechanical agitation using a Vortex or Genie[6] or using activated copper. Organic interferences can be removed with gel permeation chromatography (GPC) described in USEPA (1983(**55**)), bonded octadecyl columns (PSEP, 1997c (**268**)), high performance liquid chromatography (HPLC) described by Metro (1981 (**270**)), silica gel (PSEP, 1997c (**268**)), or alumina (USEPA, 1983) (**55**). Instrumental analyses for volatiles and semivolatiles and pesticides/PCBs are performed using gas chromatography/mass spectrophotometry (GC/MS; PSEP, 1997c (**268**)) and gas chromatography/electron capture detection (GC/ECD; Burgess and McKinney, 1997 (**271**)).

14.14 *Oil and Grease:*

14.14.1 Oil and grease tests for sediments measure material recovered that is soluble in a non-ionic solvent under acidic conditions. Oil and grease compounds are substances such as hydrocarbons, vegetable oils, animal fats, waxes, soaps, and greases. Many solvents can dissolve other substances (for example, sulfur compounds, organic dyes, and chlorophyll). Therefore, oil and grease is operationally defined by the solvent used and the analytical method used to perform the analysis. There are two basic methods used to analyze oil and grease: the gravimetric technique and the IR (infrared spectrophotometer) technique. Both are described by PSEP (1996 (**242**)).

14.15 *Total Sulfides:*

14.15.1 Total sulfides represent the combined amount of acid-soluble H_2S, HS-, and S2- in a sample. Sulfides are often measured because they are common in some sediments, particularly those that are anoxic, and they can be toxic to aquatic organisms. PSEP (1996 (**242**)) describes a method to measure total sulfides in sediments. Oxygen is removed from the sample using nitrogen gas, methyl orange and hydrochloric acid is added, and the mixture is heated. Amine solution and

[6] Genie is a trademark of Scientific Industries Inc. 70 Orville Drive, Bohemia, New York 11716.

iron chloride are added to develop a colorimetric reaction product and sample absorbance is measured spectrophotometrically. Elevated concentrations of ammonia (Sims et al. 1995a (**226**), Moore et al. 1997 (**227**)) and hydrogen sulfide (Sims et al. 1995b (**228**)) have frequently been found in deeper dredged sediment samples compared to surficial sediment samples.

14.15.2 Methods for measuring sulfides in aqueous samples include: potentiometric methods described by Practice D3976 and APHA (Method 4500, 1995 (**231**)). Sulfide ions are measured using a sulfide ion-selective electrode in conjunction with a double-junction, sleeve type reference electrode (Phillips et al. 1997 (**272**)). Potentials are read using a pH meter or a specific ion meter having a direct concentration scale for the sulfide ion. Samples are treated with sulfide anti-oxidant buffer that fixes the solution pH at a high alkaline level and retards air oxidation of sulfide ion in solution. This ensures that the sulfide measured represents total sulfides as S = ion rather than the HS- or H_2S found at lower pH values.

14.15.3 APHA (Method 4500, 1995 (**231**)) provides qualitative as well as quantitative methods to determine aqueous sulfide concentrations. Qualitative methods include the antimony test, the silver-silver sulfide electrode test, the lead acetate paper test, and the silver foil test. Quantitative methods include the photometric method, the automated photometric methylene blue colorimetric methods, and the iodometric titration method for standardizing stock solutions.

14.16 *Sediment Oxygen Demand (SOD):*

14.16.1 Sediment can exhibit significant rates of oxygen uptake attributable to either: (*1*) a benthic ecosystem supported by soluble organic substances in the water column, (*2*) naturally occurring sediments derived from aquatic plants and animals, and (*3*) detritus discharged into the water body by natural runoff. When numerical modeling is required to predict dissolved oxygen concentrations, the rate of dissolved oxygen consumed by the benthic ecosystem is defined as the sediment (benthic) oxygen demand (SOD) in g O_2/m^2-day.

14.16.2 Two approaches for measuring SOD were reviewed by Truax et al. (1995 (**273**)) including in-situ respirometry and laboratory respirometry methods. Numerous techniques have been developed for each approach. Generally, in-situ methods are considered more credible than laboratory measurements although both apply the same technique. A given amount of sediment is enclosed in a chamber with a known water volume and oxygen uptake is measured over time. The SOD rate is then calculated based on the area of the enclosed sediment, the volume of water in the chamber, and the rate of uptake.

14.16.3 In situ sediment oxygen demand measurement methods were described by Uchrin and Ahlert (1985 (**274**)). A cylindrical respirometer, a dissolved oxygen probe with stirring mechanism, and a dissolved oxygen meter were used. Ambient dissolved oxygen was measured using the probe/meter as well as by using the Winkler method (APHA, 1995 (**231**)) in the laboratory to determine the effect of respiration on total dissolved oxygen uptake. The respirometer was deployed in a level area at the bottom of the water body. Dissolved oxygen were recorded initially and at 15-min intervals thereafter to determine the SOD rate.

14.17 *Sediment Biochemical Oxygen Demand (BOD):*

14.17.1 Biochemical oxygen demand (BOD) is a measure of the dissolved oxygen consumed by microbial organisms while assimilating and oxidizing the organic matter in a sample (PSEP, 1996 (**242**)). The test is an empirical methodology in which consistent laboratory procedures are used to determine the relative oxygen uptake of environmental samples. The test measures the amount of molecular oxygen used during a specified incubation period to biochemically degrade organic material and to oxidize reduced forms of nitrogen (APHA, 1995 (**231**)).

14.17.2 Plumb (1981 (**49**)) described a method to analyze BOD in sediments using freshwater bacteria as a "seed" and buffered distilled water. PSEP (1996 (**242**)) described an alternative procedure to analyze BOD in marine sediments using marine bacteria as the "seed" and filtered, oxygenated seawater. USEPA (1987 (**43**)) methods should also be consulted.

14.18 *Sediment Chemical Oxygen Demand (COD):*

14.18.1 Chemical oxygen demand (COD) is a measure of the oxygen equivalent of organic matter content in a sample that is susceptible to oxidation by a strong chemical oxidant at elevated temperature and reduced pH. The test was devised to augment the biochemical oxygen demand test. Chemical oxygen demand can be related empirically to biochemical oxygen demand, organic carbon, or total volatile solids (PSEP, 1996 (**242**)).

14.18.2 PSEP (1996 (**242**)) described a method for analyzing sediment COD using a closed reflux/colorimetric method. Dichromate (Cr_2O_7) ions are used to oxidize organic matter to carbon dioxide and water and to provide oxygen. The dichromate ions remaining after the reaction are measured by titration and the amount of oxygen consumed is then calculated.

14.18.3 Four standards procedures for measuring COD in water are available in APHA (1995 (**231**)): the open reflux method, the closed reflux method, the titrimetric method, and the closed reflux/colorimetric method. USEPA (1983 (**55**)) methods for the colorimetric and titrimetric method are described in USEPA (1979 (**222**)). Semi-automated methods are described in USEPA (1993 (**51**)).

14.19 *Cation Exchange Capacity of Sediments:*

14.19.1 Cation exchange capacity (CEC) is a parameter that provides information relevant to metal bioavailability studies (Black, 1965 (**215**)). Cations or positively charged elements (such as calcium, magnesium, hydrogen, and potassium), are attracted to negatively charged surfaces of clay and organic matter. There is a continuous exchange of cations between sediment and water. CEC is a measure of the sediment's ability to retain cationic elements. It is also a measure of clay activity and mineralogy, which is used to calculate mineralization rates, leaching rates, and to predict interactions with contaminants. The degree of CEC is dependent on the kind and amount of suitable surfaces such as organic matter and clay. High cation exchange capacities are associated with high clay contents and high organic matter and changes in CEC are typically associated with changes in organic carbon content and pH of the sediment. Organic matter generally supplies a greater number of exchange sites than clay particles.

14.19.2 Various methods have been recommended to determine bioavailable fractions of metals in sediments (Chao and Zhou, 1983 (**275**); Crecelius et al. 1987 (**265**); Kersten and Forstner, 1987 (**276**); Di Toro et al. 1990 (**53**)). CEC can be measured by treating samples with ammonium acetate so that all exchangeable sites are occupied by NH_4+ ion, digesting the samples with sodium hydroxide during distillation, and titrating to determine the ammonium ion concentration. The amount of exchangeable cations are expressed as milliequivalents of ammonium ion exchanged (meq) per 100 g of dried sample. More detailed methods are provided in Bascomb (1964 (**277**)), Black (1965 (**215**)), Klute (1986 (**278**)), and USEPA (1986 (**223**)).

14.20 *Redox Potential (Eh) of Sediments:*

14.20.1 Redox (Eh) is a measure of the oxidation-reduction potential (ORP) of sediments. Measurements of Eh are particularly important for metal speciation and for determining the extent of sediment oxidation. Eh values below about -100 millivolts would indicate biologically important sulfide concentrations. Some trace metals form insoluble complexes with sulfides. These metal-sulfide complexes bind the metals in a form that is not bioavailable. Since free ionic metals are generally thought to possess the greatest toxicity potential, it is important to understand conditions which control binding dynamics, such as pH and Eh.

14.20.2 Potentiometric measurements of Eh using a millivolt reader can be obtained with a platinum electrode relative to a standard hydrogen electrode (Plumb, 1981 (**49**)). APHA (1995 (**231**)) does not recommend the standard hydrogen electrode as it is fragile and impractical. Instead, their method uses a silver-silver-chloride or calomel reference electrode. APHA (1995 (**231**)) recommends a graphite rather than platinum electrode for sediments. Once the Eh equilibrium is reached, the difference between the platinum or graphite electrode and the reference electrode is equal to the redox potential of the system. For a more detailed explanation on how to calculate the Eh potential see APHA (1995 (**231**)). Gonzalez (1995 (**220**)) also describes a detailed method that can be used to measure sediment Eh.

14.20.3 There are a number of problems associated with the accurate measurement and interpretation of Eh in sediments, particularly in marine sediments. Therefore, considerable attention should be paid to the use of proper equipment and techniques. Some of the problems identified by Whitfield (1969 (**279**)) and Mudroch and Azcue (1995 (**46**)) include measurement inaccuracy due to disturbance of the sediment sample during insertion of the electrode, instability and poor reproducibility of the measurements, and differential responses of platinum electrodes under different environmental conditions. A comprehensive description of the limitations of sediment Eh measurement is beyond the scope of this standard. Rather, it is recommended that published studies on the problems associated with measuring and interpreting sediment Eh be consulted before any attempt is made to measure these parameters in sediment samples (Berner, 1963 (**280**); Morris and Stumm, 1967 (**281**); Whitfield, 1969 (**279**); Tinsley, 1979 (**282**); Bates, 1981(**283**)). The recommended procedure for measuring pH and Eh in the field are described in detail in Table 15.

14.21 *Total Inorganic Carbon:*

14.21.1 Inorganic carbon has been measured as a complement to microbial activity (Bregnard et al. 1996 (**284**)), to determine the fate of an organic contaminant in biodegradation studies (West and Gonsior, 1996 (**285**)), and to determine the percent carbon unaccounted for in fate transport predictions of hydrophobic contaminants (Tye, et al. 1996 (**286**)). Often the total inorganic carbon (TIC) fraction in samples is many times greater than the TOC fraction and presents an interference in the measurement of TOC. There are several options to eliminate TIC interferences when trying to measure TOC. One option is to compensate for the IC interference by measuring total carbon (TC) and total inorganic carbon (see 14.4). The difference between the two is the TOC.

14.21.2 TIC is determined by acidifying the sample to convert the inorganic carbon (that is, carbonates, bicarbonates, and dissolved CO2) to carbon dioxide. Carbon dioxide is purged from the sample and then detected by a non-dispersive infrared detector (NDIR) calibrated to directly display the mass of carbon dioxide measured. This mass is proportional to the mass of TIC. Other instrumentation for the analysis of TIC is described in West and Gonsior (1996 (**285**)) and Tye et al. (1996 (**286**)).

14.22 *Total Volatile Solids (TVS):*

14.22.1 Total volatile solids represent the fraction of total solids that are lost on ignition at a higher temperature than that used to determine total solids. Total volatile solids are used as a crude estimate of the amount of organic matter in total solids (PSEP, 1996 (**242**)). In this regard, total volatile solids are often measured instead of, or in addition to, organic carbon content.

14.22.2 Total volatile solids are operationally defined by ignition temperature. Total volatile solids content does not always represent the organic content of a sample because some organic material may be lost at the drying temperature and some inorganic material (for example, carbonates, chlorides) may be lost at the ignition temperature. Because of the temperature dependence of total volatile solids, valid interstudy comparisons require the use of consistent drying and ignition temperatures (PSEP, 1996 (**242**)).

14.22.3 Total volatile solids measurements are generally made by igniting the sediments at 550 ± 10°C until a constant weight is achieved and reporting the percent ash-free dry weight (McLeese et al. 1980 (**135**); APHA, 1995 (**231**); Keilty et al. 1988a (**82**)). Plumb (1981) (**49**) and PSEP (1996) (**242**) describe standard methods for determining the total volatile solid content of sediments. Additional methods are provided in USEPA (1987) (**43**).

14.23 *Dissolved Organic Carbon in Pore Water:*

14.23.1 Dissolved organic carbon (DOC) often consists of humic substances, and is the fraction of the organic carbon pool that is dissolved in water and passed through a 0.45 µm glass fiber filter. DOC is an indicator of the chemically reactive organic fraction and accurately measures the dissolved organic load. Sediment pore waters can be rich in humic acids. Fifty to

TABLE 15 General Procedures for Measurement of Eh in Bottom Sediments (from Murdoch and Azcue 1995 (46))

Equipment and solutions used in the measurements:
A portable, battery-operated pH/Eh meter, batteries, and a power cord for recharging the meter.
Combination glass and platinum electrodes or other electrodes suitable for the measurements.
Plastic test-tube-shaped containers or other containers for storing the electrodes in solutions during transport in the field.
Commercially-available or laboratory-prepared pH buffer solutions (pH 4 and 7) in plastic bottles with lids.
Freshly-prepared solution for calibration of Eh electrode in a plastic bottle with a tight lid.
Freshly-prepared solution of saturated potassium chloride for storage of the electrodes.
Other solutions necessary for proper functioning of electrodes as outlined by manufacturers.
Deionized water and wash bottle for storing and rinsing the electrodes between measurements.
Several small and larger plastic beakers for holding solutions, rinsing electrodes, etc..
Support stands, rods, clamps to secure electrodes in solutions and during measurements.
Large plastic containers for storage and transport of used buffers and Eh-calibration solutions.
Notebook and pens, soft paper tissue.
Preparation of equipment before the field trip:
Check batteries of the portable pH/Eh meter and replace/recharge them, if necessary.
Prepare calibration solutions.
Check and test the pH and Eh electrodes.
Mark the electrodes vertically at desired intervals for insertion into the sediment samples.
Store the electrodes according the manufacturers instructions.
Pack all equipment for transport to the field and take spare electrodes if available.
Measurements in the field:
Allocate a space where measurements will be carried out. Within this space, all equipment should be assembled, checked for proper functioning, and prepared for measurement of the first sample.
Place grab sampler and sediment cores with recovered sediment in such a way that they will remain steady without disturbing the sediment samples during the measurements.
Insert electrodes carefully into the undisturbed sediment samples to avoid any air. contamination, particularly around the Eh electrode. Care must be taken not to generate any open space between the electrode and the sediment. Proper insertion of the electrode without disturbing the sediment is the most important step in measuring the Eh.
Insert electrodes into the sediment to the depth marked. Switch the pH/Eh meter to the pH scale and the value recorded within 1 minute after inserting the electrode into the sample. Switch the meter to the mV scale for recording the Eh value. The potential usually drifts considerably over the first 10 to 15 min, and then stabilizes. After stabilization, record the mV value. In measuring Eh of sediments from waters with low ionic strength, such as most freshwater bodies, it is recommended to "acclimatize" the electrodes in the water prior to measurement, particularly the electrodes that were stored in saturated potassium chloride solution. This will reduce the drifting of the potential after inserting the electrode into the sediment.
Remove both electrodes, wash them with distilled water to remove all adhering sediment particles, and dry them gently with a soft paper tissue.
Calibrate the electrodes after each five measurements. The electrodes may need less frequent calibration if pH and Eh are being measured in a sediment core.

90 % of the pore water DOC can be colloidal which is a significant factor because organic chemicals will preferentially partition to pore water DOC (Resendes et al. 1992 (**287**); Burgess 1996 (**154**)). Dissolved organic carbon (DOC) often consists of humic substances, and is the fraction of the organic carbon pool that is dissolved in water and passed through a 0.45 µm glass fiber filter. DOC is an indicator of the chemically reactive organic fraction and accurately measures the dissolved organic load. Sediment pore waters can be rich in humic acids. Fifty to 90 % of the pore water DOC can be colloidal which is a significant factor because organic chemicals will preferentially partition to pore water DOC (Resendes et al. 1992 (**287**); Burgess 1996 (**154**)).

14.23.2 Hermann (1996 (**48**)) and Gilek et al. (1996 (**288**)) measured DOC using a TOC apparatus and infrared detection of CO_2. Borga et al. (1996 (**289**)) measured DOC using atomic emission spectrometry (ECP-AES). The APHA (Method 5310, 1995 (**231**)) methods for total organic carbon that can be applied to the measurement of DOC are (*a*) the combustion-infrared method; (*b*) the persulfate-ultraviolet oxidation method; and (*c*) the wet-oxidation method. Adjustments for inorganic carbon interference may be required.

14.24 *Alkalinity and Hardness of the Pore Water (Freshwater Sediments):*

14.24.1 Alkalinity is defined as the acid-neutralizing (that is, proton-accepting) capacity of water. It is the sum of all the titratable bases, and a measure of the quality and quantity of constituents in the pore water that result in a shift in the pH toward the alkaline side of neutrality. The measured value may vary significantly with the pH end-point used. Studies have shown that effects of certain contaminants such as metals are influenced by alkalinity as it alters speciation and bioavailability.

14.24.2 APHA (1995 (**231**)) recommends a color-change titration method to measure alkalinity which is also described by Test Method D1067. The sample is titrated with standard alkali or acid to a designated pH and the endpoint is determined electrometrically or by the color change of an internal standard. In addition, Test Method D1067 describes two additional methods: (*1*) a titration curve is developed to identify inflection points, a standard acid or alkali is added to the sample by small increments and pH is recorded after each addition, and the total volume of acid or alkali is plotted against the observed pH values; and (*2*) pH is determined, standard acid is added to lower the pH to 4.0 or less, the solution is boiled with hydrogen peroxide, and titrated, while hot, to the phenolphthalein endpoint or, when cooled, electrometrically with standard alkali to pH 8.2, the desired endpoint. The color-change titration method is most commonly used.

14.24.3 Hardness is the concentration of metallic cations, with the exception of alkali metals, present in water samples. Generally, hardness is a measure of the concentration of calcium and magnesium ions in water. Hardness is usually expressed as a calcium carbonate equivalent in mg/L. Like

alkalinity, hardness alters speciation and bioavailability of certain contaminants particularly many metals.

14.24.4 APHA (Method 2340, 1995 (**231**)) describes two methods to measure hardness: (*1*) the calculation method and (*2*) the EDTA titrimetric method. Test Method D1126 describes the APHA (1995 (**231**)) EDTA titrimetric method. Calcium and magnesium ions in water are sequestered by the addition of EDTA. The endpoint of the reaction is measured by means of Chrome Black T3, which is red in the presence of calcium and magnesium and blue when both are sequestered. APHA recommends the calculation method because it is more accurate. The method uses direct determinations of calcium and magnesium to determine hardness. The APHA EDTA titration method is most often used.

15. Quality Assurance

15.1 *General Procedures:*

15.1.1 Quality assurance activities provide a formalized system for evaluating the technical adequacy of sample collection and laboratory analysis activities. These quality assurance activities begin before samples are collected and continue after laboratory analyses are completed, requiring ongoing coordination and oversight. The quality assurance program should integrate management and technical practices into a single system to provide data that are sufficient, appropriate, and of known and documented quality.

15.1.2 Developing and maintaining a quality assurance (QA) program requires an ongoing commitment by project management and also includes the following: (*1*) appointment of a quality assurance officer with the responsibility and authority to develop and maintain a QA program, (*2*) preparation of a Quality Assurance Project Plan with Data Quality Objectives, (*3*) preparation of written descriptions of Standard Operating Procedures (SOPs) for sediment sampling and manipulations, instrument calibration, sample chain-of-custody, laboratory sample tracking system, and (*4*) provision of adequate, qualified technical staff and suitable space and equipment to provide reliable data. Program specific guidance for developing and maintaining a QA program should be followed as appropriate. Examples of program guidance for developing a quality assurance program can be found in USEPA (1994 (**78**); 1995 (**254**); 2000d (**30**)), PSEP (1997a) (**34**), WDE (1995) (**29**), and USEPA/USACE (1991 (**33**), 1998 (**35**)).

15.1.3 Quality control (QC) practices consist of more focused, routine, day-to-day activities carried out within the scope of the overall QA program. QC is the routine application of procedures for obtaining data that are accurate (precise and unbiased), representative, comparable, and complete. QC procedures include activities such as identification of sampling and analytical methods, calibration and standardization, and sample custody and record keeping. Audits, reviews, and complete and thorough documentation are used to verify compliance with predefined QC procedures. Project-specific QA plans (QAPP; 15.3) provide a detailed plan for activities performed at each stage of the study and outline the data quality objectives that should be achieved. Through periodic reporting, QA activities provide a means to track progress and milestones, performance of measurement systems, and data quality. A complete project-specific QA/QC effort has two major components: a QA program implemented by the responsible organization (that is, the data user) and QC programs implemented by the parties responsible for collection and analyses (that is, the data generators).

15.2 *QA/QC Procedures for Sediment Collection and Manipulation:*

15.2.1 To establish the appropriateness of the sample collection procedure for sample integrity and to establish that data of suitable quality, a program of scheduled field QC samples, such as field replicates (duplicates, splits, field spikes), field blanks (rinsate equipment), bottle, trip, and background (upgradient) samples. All field QC samples should be handled exactly as the sediment samples and should be treated as blind samples so as to minimize bias in the analysis. A random portion of the samples should also be analyzed by a third party to evaluate the primary laboratory's performance. QC replicates (duplicates, splits) should be collected for analysis by the primary laboratory to determine analytical variability (USEPA 1995 (**254**)).

15.2.2 The procedures for sediment manipulations described in Section 11 should maintain the sample in a chemical condition as similar as possible to that at the time of collection. QA procedures are established to assure that SOPs are followed and that contamination is neither introduced to nor lost from the manipulated sample. For example, samples to be analyzed for trace metals should not come in contact with metal surfaces (except stainless steel). Sample tracking sheets should document date, time, and investigator each time a sample is removed from storage or replaced back into storage. Specific manipulation procedures should follow established SOPs that minimize chemical alteration of the sample (excepting chemical spiking), maintain sediment physical properties, and include replication and blank samples.

15.3 *The Quality Assurance Project Plan (QAPP):*

15.3.1 The Quality Assurance Project Plan (QAPP) is a project-specific document that specifies the data quality and quantity requirements needed for the study as well as all procedures that will be used to collect, analyze, and report those data.

15.3.2 The QAPP uses input from the sampling design derived from the Data Quality Objectives Process (see Section 9, 9.6, and USEPA, 2000a (**12**)) to specify the above elements. This Plan should be reviewed by an independent person (for example, quality assurance officer or staff member not involved in the project directly) for accuracy and completeness. A key element of a QAPP is Standard Operating Procedures (see 15.4). Further information on preparing a QAPP and resources necessary can be found in USEPA (2000e (**290**)).

15.4 *Standard Operating Procedures*—Standard operating procedures are written descriptions of routine methods and should be provided for all methods used. A large number of field and laboratory operations can be described in standard operating procedures. General types of procedures that benefit from standard operating procedures include field measurements ancillary to sample collection (for example, water

quality measurements or mixing model input measurements); chain-of-custody, sample handling, and shipment; and routine analytical methods for chemical analyses and toxicological analyses. Standard operating procedures are used to establish that all persons conducting work are following the same procedures and that the procedures do not change over time. All personnel should be familiar with the standard operating procedures before work is initiated. Deviations from standard operating procedures might affect data quality and integrity. If it is necessary to deviate from approved standard operating procedures, these deviations need to be documented and approved through an appropriate chain-of-command.

15.5 *Sediment Sample Documentation*—Bound field logbooks should be used for the maintenance of field records. All entries should be dated and time of entry recorded. All aspects of sample collection and handling as well as visual observations should be documented in the field logbooks. Documentation should be recorded in pre-numbered bound notebooks using indelible ink pens in sufficient detail so that decision logic may be traced back, once reviewed.

15.6 *Sample Tracking Documentation:*

15.6.1 Samples delivered to the laboratory should be accompanied by a chain-of-custody record that includes the name of the study, location of collection, date and time of collection, type of sample, sample name or number, number of containers, analysis required, and the collector's signatures. When turning over possession of samples, the relinquisher and the receiver sign, date and record the time on the record sheet. The record sheet allows the transfer of a group of samples at one time. When the laboratory takes possession of the samples, each should be assigned a unique laboratory identification designation. This will provide a consistent system for tracking within the laboratory. If the samples arrive at the laboratory when designated personnel are not there to receive them, the samples are put into a secure location and the transfer is conducted when the appropriate personnel are present.

15.6.2 Upon arrival at the laboratory, samples are inspected for condition and temperature, and sample container labels are verified against the chain-of-custody record or sample tracking form. Sample information is entered on laboratory log-in data sheets used to maintain information regarding sample: receipt, shipping, collection date, and storage. To allow for accurate identification of samples, information contained on sample tracking forms needs to match identically with information contained on the sample container labels. The tracking form lists both the collector's and the laboratory's identification designations. Verified tracking forms are signed by the laboratory personnel with date and time in ink. Missing or compromised samples (for example, inappropriate preservation to maintain integrity, inappropriate containers, and unlabeled or mislabeled containers) are documented on the tracking forms.

15.6.3 When samples are removed from storage, the sample tracking form accompanies it and documents date, time, and investigator associated with any manipulations. The manipulation type is noted on the form in detail or by reference to an approved laboratory SOP. Any deviations from the SOP are also noted. Should the sample be modified in such a way that additional subsamples are created, additional tracking forms need to be created.

15.7 *Record Keeping*—Proper record keeping is essential to the scientific defensibility of sediment sampling and manipulation. A separate file should be maintained for each sampling or manipulation event or closely related events. This file should contain field logs, chain-of-custody forms, sample tracking forms, storage records, and any QA/QC documentation and records. Original documentation should be signed and dated by the originator.

15.8 *QA Audits*—In addition to the QA/QC procedures conducted on a routine basis, quality audits (that is, performance and quality systems audits) might be conducted. Performance audits refer to independent checks to evaluate the quality of data produced during testing. There are three types of performance audits: sampling, test, and data processing. These audits are independent of normal quality control checks performed by the operator. A systems audit is an on-site inspection and review of the quality assurance system. The systems audit is performed to verify that the organization is following the policies and procedures described in its QA/QC plan and in appropriate SOPs. Systems audits are performed by an auditor typically from an accrediting body.

15.9 *Corrective Action (Management of Non-conformance Events):*

15.9.1 The QA Officer and the responsible manager are responsible for reviewing the circumstances of all instances of occurrence of nonconformities, to determine whether corrective action should be taken. The manager is responsible for determining if new samples are required, if the customer should be notified, if additional testing is necessary, or whether the results should be confirmed. A good communication plan is invaluable in helping to identify interactions among labs, clients, and agencies during corrective actions.

15.9.2 Corrective action might take two forms: that of addressing technical problems associated with project activities and that of addressing QA/QC infractions based upon performance. Technical problems in meeting project objectives may range in magnitude from failure to meet minor procedural requirements, to major problems associated with inappropriate methods or data loss.

15.9.3 Established procedures for corrective action of minor technical problems are often included in the SOPs for cases where performance limits or acceptance criteria have been exceeded. On-the-spot corrective actions are noted on data sheets. Major or recurrent QA/QC problems which require long-term corrective action, such as modification of SOPs, are reported. Depending upon the nature and severity of the problem, an approach might be developed. Any corrective action is documented by management.

15.9.4 Infractions of QA/QC policies by staff are identified and addressed by the management. Minor infractions are corrected through additional training or closer supervision. Major or recurrent infractions are corrected through reassignment of technical personnel.

15.9.5 Corrective actions relative to sample collection and manipulation may include, but are not limited to, review of the

data and calculations, flagging or qualification of suspect data, or possible re-sampling. A review that provides a preliminary check of all "out of limit" events is performed as soon as the data for a given parameter or test is tabulated and verified for accuracy. "Out of limit" events are flagged to determine whether new samples are required.

16. Report

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

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

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

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

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

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

17. Keywords

17.1 basket samplers; benthic macroinvertebrates; characterization; collection; interstitial water; manipulation; multiplate samplers; pore water; sediment; sediment grab samplers; spiking; storage; stream net sampling devices; toxicity; transport

ANNEX

(Mandatory Information)

A1. DESCRIPTION OF SAMPLERS USED TO COLLECT SEDIMENT OR BENTHIC INVERTEBRATES

A1.1 Significance

A1.1.1 This annex describes sampling devices that can be used to collect sediment or benthic macroinvertebrates. These include grab sampling devices (Table A1.1) and stream-net sampling devices (Table A1.2). This annex also covers methods for deploying basket samplers and multiplate samplers for collecting benthic macroinvertebrates.

A1.1.2 This annex was developed by consolidating information from the following ASTM standards that were subsequently withdrawn when the standard was approved.

D4387-84 (2002) Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates

D4556-85 (2002) Guide for Selecting Stream-Net Sampling Devices for Collecting Benthic Macroinvertebrates

D4342-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ponar Grab Sampler

D4343-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler

D4344-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Smith-Mcintyre Grab Sampler

D4345-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Van Veen Grab Sampler

D4346-84 (1997) Practice for Collecting Benthic Macroinvertebrates with Okean 50 Grab Sampler

D4347-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Shipek (Scoop) Grab Sampler

D4348-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Holme (Scoop) Grab Sampler

D4401-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Petersen Grab Sampler

D4407-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Orange Peel Grab Sampler

D4557-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Surber And Related Type Samplers

D4558-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Drift Net

E1468-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Basket Sampler

E1469-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Multiplate Sampler

A1.2 Terminology Specific to this Annex

A1.2.1 *benthos*—the community of organisms living in or on the bottom or other substrate in an aquatic environment.

A1.2.2 *grab*—any device designed to "bite" or "scoop" into the bottom sediment of a lake, stream, estuary, ocean, and similar habitats to sample the benthos. Grabs are samplers with jaws that are forced shut by weights, lever arms, springs, or cables. Scoops are grab samplers that scoop sediment with a rotating container.

A1.2.3 *habitat*—the place where an organism lives, that is, mud, rock, shoreline, etc.

A1.2.4 *macroinvertebrates*—benthic or substrate dwelling organisms visible to the unaided eye and retained on a U.S. Standard No. 30 (0.595-mm mesh openings) sieve. The standard sieve opening for marine benthic fauna is 1.0 mm, U.S. Standard No. 18 sieve. Examples of macroinvertebrates are aquatic insects, macrocrustaceans, mollusks, annelids, roundworms, flatworms, and echinoderms.

TABLE A1.1 Classification of Grab Sampling Devices for Collecting Benthic Macroinvertebrates

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Ponar Grab	Freshwater lakes, rivers, and estuaries, reservoirs	Hard sediments, except hard clay; some-what less efficient in softer sediments	Sample area 523 cm^2; efficient and versatile; not entirely adequate for deep burrowing organisms in soft sediments; quantitative and qualitative sampling obtained; sediment inhabiting macro-invertebrates	Better penetration than other grabs; side plates and screens pre-vent washout and shock wave that accompany other grabs	Requires boat, winch, and cable; jaws can be blocked and part of sample lost	Better for quantitative sampling than Petersen grab	Brinkhurst (291, 292) Elliot and Drake (293) Elliott and Tullet (294) Flannagan (295) Howmiller (296) Hudson (297) Lewis, Mason, Weber (298) Powers and Robertson (299) Weber (300) Klemm et al. (301)
Petite	Freshwater lakes, rivers, and estuaries, reservoirs	Hard sediments, except hard clay; some-what less efficient in softer sediments	Sample area 232 cm^2; efficient and versatile; not entirely adequate for deep burrowing organisms in soft sediments; sediment inhabiting macro-invertebrates	Better penet-ration than other grabs; side plates and screens pre-vent washout and shock wave that accompany other grabs; can be operated by hand	Jaws can be blocked and part of sample lost; insufficient in swiftly moving water to 1 m/s velocity		Klemm et al.(301) Merrit et al. (302)Gerritsen et al. (303)
Ekman Grab	Freshwater lakes, reservoirs, where there is little current; usually small bodies of water	Soft sediments only	Sample area 232 cm^2; efficient in soft sediments; extra weights can be used for deeper penetration; quantitative and qualitative obtainable; sediment inhabiting macroinverte-brates	Can be operated by hand; can be operated in shallow, sand or mud bottom streams; comes in a range of sizes	Jaws can fail to penetrate; only partial cylinder cut from substrate, small surface area coverage jaws can be blocked and part of sample lost; inefficient in deep water or moderate to strong currents		Beatties (304) Burton and Flannagan (305) Ekman (306,307) Elliott and Drake (293) Elliott and Tullett (294) Flannagan (295) Howmiller (296) Hudson (297) Lanz, (308) Lewis, Mason, Weber (298) Lind (309) Milbrink and Wiederholm (310) Rowe and Clifford (311)Lewis et al.(312) Klemm et al. (301)Merritt et al. (302)Gerritsen et al.(303)
Tall	Same as above	Same as above	Sample area 232 cm^2 Same as above	Same as above	Same as above		Paterson and Fernando (313) Schwoerbel (314)
Large	Same as above	Same as above	Sample area 523 cm^2 Same as above	Same as above	Same as above		Rawson (315) Welch (316) Weber (300)
Extra Large	Same as above	Same as above	Sample area 929 cm^2	Same as above	Same as above		

A1.3 Significance and Use

A1.3.1 Grab samplers for collecting sediments or benthic macroinvertebrates: Qualitative and quantitative samples of macroinvertebrates inhabiting sediments or substrates are often collected using a grab sampler. In view of the advantages and limitations regarding the penetration of the sediment by many

TABLE A1.1 *Continued*

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Petersen Grab	Freshwater lakes, reservoirs; adaptable to rivers, estuaries, and oceans	Sand, gravel, mud, clay	Sample penetration limited sample area from 0.06 to 0.099 m^2; sediment inhabiting macroinverte-Brates	Gives reasonable quantitative samples when used carefully; comes in a range of sizes	Fairly heavy; need boat and power winch; jaws maybe blocked by sand, etc.; inadequate for deep burrowing organisms; questionable value for strictly quantitative samples; hard to use in adverse weather conditions	Least preferred grab sampler	Barnes (317) Birkett (318) Brinkhurst, (291,292) Davis (319) Edmondson and Winberg (320) Davis (319) Elliott and Tullett (294) Holme and McIntyre (321) Hudson (297) Howmiller (296) Lewis, Mason, Weber (298) Lind (309) Petersen (322) Thorson (323) Welch (316) Weber, (300) Petersen and Boysen Jensen (324)Klemm et al.(301)
Smith-McIntyre Grab	Marine and estuaries; adaptable to large rivers, lakes	Sand, gravel, mud, clay, and similar substrates	Sample area limited to 0.1 m^2 with approximately 4 cm deep in hard sand; reasonably quantitative; sediment inhabiting macro-invertebrates	Reasonable quantitative samples; the trigger plates provide added leverage essential to its penetration of substrate	Heavy; need boat and power winch; spring-loaded jaws, hazardous; jaws can be blocked; inadequate for deep burrowing organisms	Widely acceptable sampling device for use in marine and estuary habitats	Carey and Heyamoto (325) Carey and Paul (326) Elliott and Tullett (294) Holme (327,328) Hopkins (329) Hunter and Simpson (330) McIntyre (331) Smith and McIntyre (332) Tyler and Shackley (333) Wigley (334) Word (335) Klemm et al.(301)
Van Veen Grab	Marine and estuaries, adaptable to freshwater areas	Sand, gravel, mud, clay, and similar substrates	Sample area 0.1 m^2 and 0.2 m^2; reasonable penetration; to depth of approximately 5 to 7 cm; sediment inhabiting macroin-vertebrates	Jaws close tighter than Petersen grab; samples most sediment types; comes in a range of sizes	Need large boat, power winch and cable line; blockage of jaws may cause sample loss; not useful for deep burrowing organisms	Limited application	Barnes (317) Beukema (336) Birkett (318) Elliott and Drake (293) Elliott and Tullet (294) Holme (327,328) Lassig (337) Longhurst (338) McIntyre (331) (339) Nichols and Ellison (340) Schwoerbel (314) Ursin (341) Wigley (334), Word (342,343) Word (335) Klemm et al(301)

grab samplers and their closing mechanisms, it is not possible to recommend any single instrument as suitable for general use. However, the Petersen grab is considered the least effective bottom grab sampler and, therefore, has limited application. The type and size of the grab sampler or device selected for use will depend on such factors as the size of boat, hoisting gear available, the type of substrate or sediment to be sampled, depth of water, current velocity, and whether sampling is in sheltered areas or in open waters of large rivers, reservoirs, lakes, and oceans. A great variety of instruments have been described and choice of a grab sampler will depend largely on what is available, what is suitable for the sampling

TABLE A1.1 *Continued*

Grab Sampling Device	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Orange-Peel Grab	Marine waters, deep lakes	Sandy substrates, cobble, rubble stone	Sample area 0.025 m^2; penetration depth about 18 cm; qualitative sampler, not a satisfactory quantitative sampler; should not be used in critical quantitative work that is to be compared with results from other sampling areas; sediment inhabiting macro-invertebrates	Comes in a range of sizes	Need large boat, powered which and cable line; blocking of jaws may cause sample loss	Limited application; reconnaisance sampling only	Briba and Reys (344) Elliott and Tullett (294) Hartman (345) Hopkins (329) Merna (346) Packard (347) Reish (348) Thorson (323) Word (342)Klemm et al(301)
Okean 50 Grab	Marine, estuarine, also large rivers	Sand, gravel, mud, clay, similar substrates	Sample area 0.25 m^2; should be lowered slowly for quantitative work; moderately deep penetration in hard sand; better for quantitative sampling than Petersen grab; sediment inhabiting macroin-vertebrates	Moderately deep penetration in hard sand; gauze covered window at top of each bucket to allow water to escape while grab is closing; offer some resistance to swift currents; lowering of grab desirable for deep sea sampling; may also have hinged doors instead of screened windows; rapid rates of lowering are possible; comes in a range of sizes	Heavy; requires large boat, powered winch and cable line; jaws may be blocked and sample lost; not entirely adequate for deep burrowing organisms; should be lowered slowly for quantitative sampling		Elliott and Tullett (294) Holme (327,328) Holme and McIntyre (321) Lisitsin and Udintsen (349) Zhadin (350)
Shipek Grab	Estuarine areas, also large freshwater lakes	Sand, gravel, mud, clay, and similar substrates	Sample area 20 cm^2, approximately 10 cm deep at left; sediment inhabiting macroinverte-brates	Scoop type sampler	Heavy; requires boat, powered winch and line; should be lowered on a near vertical line; inadequate for deep burrowing organisms; sampled area may be rather small for quantitative work	Limited application	Barnes (317) Elliott and Tullett (294) Flannagan (295) Holme (327,328) Holme and McIntyre (321)
Holme Grab	Marine, estuarine areas, deep lakes	Sand, gravel, mud, clay, and similar substrates	Sample area 0.05 m^2, approximately 15 cm. in hard sand, etc., sediment inhabiting macroinverte-Brates	Scoop type sampler; comes with a single scoop or double scoop	Heavy; requires boat, powered winch and line; springs of scoop may be difficult to reset; inadequate for deep burrowing organisms	Limited application	Barnes (317) Elliott and Tullett (294) Holme (327,347) (39) Holme and McIntyre (321) Thorson (323)

area, and what can be obtained without difficulty. This annex describes the following grab samplers for collecting sediment or benthic macroinvertebrates: (*1*) Ponar, (*2*) Ekman, (*3*) Petersen, (*4*) Smith-McIntrye, (*5*) Van Veen, (*6*) Orange-Peel,

TABLE A1.2 Classification of Stream-Net Samplers for Collecting Benthic Macroinvertebrates

Stream-Net Samplers	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Surber sampler	Shallow, flowing waters, depth recommended	Mud, sand, gravel, or rubble substrates	Depends on experience and ability of user; area sampled 0.1 m^2; performance depends on current and substrate; size of macroinvertebrates collected depends on mesh size; variety of mesh sizes may be used.	Easily transported or constructed; samples a unit area; partial screen enclosure	Does not produce quantitative samples consistently; clogging with sand or algae; difficult to set in some substrate types, that is, large rubble; cannot be used efficiently in still or deep water.	Can be modified to fit difficult situations.	Elliot and Tullett (351)Ellis and Rutter (352) Lane (353) Merritt, Cummins, and Resh (354) Needham and Usinger (355) Pollard and Kinney (356) Rutter and Ettinger (357) Resh (358) Rutter and Poe (359) Surber (360) (361) Welch (362) Kroger (363)Klemm et al (301)
Portable invertebrate box sampler	Same as above	Same as above	Same as above	Same as above; completely enclosed; limits escape of organisms; stable platform; can be used in weed beds.	Same as above	Same as above	Resh, et al (364)
Hess sampler	Same as above	Same as above	Same as above	Same as above; completely enclosed; limits escape of organisms; can be used in weed beds.	Same as above	Same as above	Canton and Chadwick (365) Elliott and Tullett (351) Hess (366) Merritt, Cummins, and Resh (354) Pollard and Kinney (356) Resh (358) Usinger (367) Welch (362) Resh, et al (364) Klemm et al.(301)
Hess stream bottom sampler	Shallow, flowing waters, depth recommended	Mud, sand, gravel, or rubble substrates	Depends on experience and ability of user; area sampled 0.1 m^2; performance depends on current and substrate; size of macroinvertebrates collected depends on mesh size; variety of mesh sizes may be used.	Easily transported, or constructed; samples a unit area completely enclosed; limits escape of organisms; can be used in weed beds.	Does not produce quantitative samples consistently; clogging with sand or algae; difficult to set in some substrate types that is, large rubble; cannot be used efficiently in still or deep water.	Can be modified to fit difficult situations	
Stream-bed fauna sampler	Same as above	Same as above	Same as above	Same as above	Same as above	Same as above	
Drift nets	Flowing rivers and stream	Drifting benthic macroinvertebrates from all substrate types.	Effective in collecting all taxa which drift in the water column; performance depends on current velocity and sampling period; size of macroinvertebrates collected depends on mesh size used.	Low sampling error; less time, money, effort; collects macroinvertebrates from all substrates; usually collects more taxa.	Unknown where organisms come from; terrestrial species may make up a large part of sample in summer and periods of wind and rain.	Limited application	Allen (368) Allan and Russek (369) Bailey (370) Berner (371) Chaston (372) Clifford (373) (374) Cushing (375,376) Dimond (377) Edington (378) Elliott (379,380,381,382, 383,384) Ferrington (385) Hales and Gaufin (386) Hildebrand (387) Holt and Waters (388) Hynes (389) Klemm

TABLE A1.2 *Continued*

Stream-Net Samplers	Habitat Sampled	Substrate Type Sampled	Effectiveness of Sampling Device; Taxa Sampled	Advantages	Limitations	Preference or Recommendation	Selected Literature
Drift nets	Flowing rivers and stream	Drifting benthic macroinverte-brates from all substrate types.	Effective in collecting all taxa which drift in the water column; performance depends on current velocity and sampling period; size of macroinvertebrates collected depends on mesh size used.	Low sampling error; less time, money, effort; collects macroinvertebrates from all substrates; usually collects more taxa.	Unknown where organisms come from; terrestrial species maymake up a large part of sample in summer and periods of wind and rain.	Limited application	Keefer and Maughan **(390)** Larimore **(391)** Larkin and McKone **(392)** Lehmkuhl and Anderson **(393)** McLay **(394)** Merritt, Cummins, and Resh **(354)** Minshall and Winger **(395)** Modde and Schulmbach **(396)** Muller **(397,398)** Mullican, Sansing, and Sharber **(399)** Mundie **(400,401)** Pearson and Franklin **(402)** Pearson and Kramer **(403,404)** Pearson, Kramer, and Franklin **(405)** Pfitzer **(406)** Radford and Hartland-Rowe **(407)** Reisen and Prins **(408)** Resh **(358)** Resh, et. al **(364)** Spence and Hynes **(409)** Tanaka **(410)** Tranter and Smith **(411)** Waters **(412,413, 414,415,416, 417,418,419, 420,421)** Weber **(422)** Wilson and Bright **(423)** Winner, Boesel, and Farrell **(424)** Wojtalik and Waters **(425)**

(*7*) Okean 50, (*8*) Shipek, and (*9*) Holme. Tables A1.3 and A1.4 describe advantages and disadvantages of commonly used grab or core samplers.

A1.3.2 Stream-net sampling devices for collecting benthic macroinvertebrates: Stream-net samplers are used to collect macrobenthos inhabiting a wide range of habitat types from shallow flowing streams or shallow areas in rivers. The stream-net devices (Surber, portable invertebrate box, Hess, Hess stream bottom, and stream-bed fauna samplers) are unit area samplers used for collecting benthic organisms in certain types of substrates. These devices are hand operated and permit collections of qualitative or reasonably quantitative samples of benthic macroinvertebrates from flowing shallow waters. They are used to obtain quantitative estimates of the standing crop, for example, biomass, number of individuals and number of taxa of benthic macroinvertebrates per unit area of stream bottom. Drift nets are another type of qualitative and quantitative sieving device that are useful for collecting benthic macroinvertebrates that either actively or passively enter the water column from all types of substrates in flowing waters. These devices are used to determine the drift of benthic organisms from a variety of substrate types at one time.

A1.3.3 Basket and multiple-plate sampling devices for collecting benthic macroinvertebrates: Basket samplers are used to collect qualitative and quantitative samples from lentic and lotic waters containing benthic macroinvertebrates living on various types of substrates. The materials used in the basket sampler are natural or artificial materials of various compositions and configurations. The device is placed in water for a predetermined exposure period and depth for the colonization of macroinvertebrate communities. Multiple-plate samplers consist of artificial substrate surfaces (tempered hardboard or ceramic plates) for colonization by aquatic organisms. Their uniform shape and texture compared to natural substrates greatly simplifies the problem of sampling relative to basket samplers. Physical factors such as stream velocity and depth may variably affect the degree of colonization. The sampling method is selective for drifting organisms (biased for insects) and for those that preferentially attach to or live on hard surfaces.

A1.4 General Hazards

A1.4.1 Inspect samplers for mechanical defects prior to use.

A1.4.2 Exercise caution when handling the samplers.

TABLE A1.3 Advantages and Disadvantages of Commonly Used Grab Samplers

NOTE 1—Modified from Klemm et al., 1990 (**31**); Environment Canada, 1994(**2**); PSEP, 1997a(**34**) ; WDE, 1995(**29**); USEPA 2001 (**1**).

Device	Use	Sample Depth, cm	Sample Volume, L^3	Advantages	Disadvantages
Orange Peel	Marine waters, deep lakes	0 to 18	10 to 20	Comes in a range of sizes	Need large boat, powered winch and calbe line Blocking of jaws may cause sample loss
Smith-McIntyre	Deep lakes, rivers and estuaries	0 to 4 (in deep sand)	10 to 20	Reasonable quantitative samples The trigger plates provide added leverage essential to its penetration of substrate	Heavy, need boat and power winch Spring loaded jaws, hazardous Inadequate for deep burrowing organisms
Birge-Ekman, small	Lakes and marine areas; soft sediments, silt and sand	0 to 10	≤ 3.4	Handles easily without winch or crane Can be adapted for shallow water use Good for soft sediments, sand and silt Allows subsampling	Restricted to low current due to light weight and messenger activation May exceed target penetration depth Subsampling may be restricted by size of top flaps
Birge-Ekman, large	Lakes and marine areas; soft sediments, silt and sand	0 to 30	≤ 13.3	Can be adapted for shallow water use Good for soft sediments, sand and silt Allows subsampling	Restricted to low current conditions Penetration depth can exceed desired level due to weight of sampler Heavy; requires winch
PONAR	Deep lakes, rivers and estuaries; useful on sand, silt or clay	0 to 10	7.25	Most universal grab sampler Adequate on most substrates Large sample obtained intact, permitting subsampling Good for coarse and firm bottom sediments	May not close completely, resulting in sample loss Metal frame may contaminate sample Heavy; requires winch
PONAR, petite	Deep lakes, rivers and estuaries; useful on sand, silt or clay	0 to 10	1.0	Adequate for most substrates that are not compacted	May not penetrate sediment to desired depth, especially in consolidated sediments. Susceptible to incomplete closure and loss of sample. Requires more casts to obtain sufficient sample if many analyses needed.
Van Veen	Deep lakes, rivers and estuaries; useful on sand, silt or clay; effective in marine environments in deep water and strong currents	0 to 30	18 to 75	Adequate on most substrates that are not compacted Large sample obtained intact, permitting subsampling Available in stainless steel	May not close completely, resulting in sample loss May close prematurely in rough waters Metal frame may contaminate sample Heavy; requires winch
Modified Van Veen (for example, "Ted-Young grab")	Lakes and marine areas	0 to 15	≤ 18.0	Fluorocarbon plastic liner can help avoid metal contamination Screened bucket cover helps reduce bow wave effects	Requires winch Relatively expensive
Petersen	Deep lakes, rivers and estuaries; useful on most substrates	0 to 30	9.45	Provides large sample Penetrates most substrates	Shock wave from descent may disturb fine-grained sediment Lacks lid cover to permit subsampling May not close completely, resulting in sample loss Metal frame may contaminate sample Restricted to low current conditions May exceed target penetration depth
Shipek	Used primarily in marine waters and large inland lakes and reservoirs; not useful for compacted sandy clay or till substrates	0 to 10	3.0	Sample bucket opens to permit subsampling Retains fine-grained sediments effectively	Metal frame may contaminate sample Heavy; requires winch II.Can result in the loss of the topmost 2-3 cm of very fine, unconsolidated sediment
Mini Shipek	Lakes, useful for most substrates that are soft	0 to 3	0.5	Handles easily without winch or crane from most platforms	Requires vertical penetration Samples small volume May lose fine-grained sediment May close prematurely

A1.4.3 Clean samplers between use (see 10.4).

A1.5 Descriptions of Samplers

A1.5.1 *Ponar Grab Sampler:*

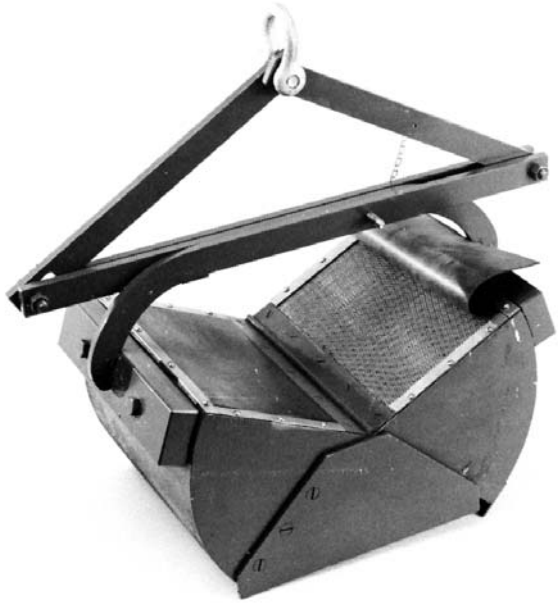
A1.5.1.1 A Ponar Grab Sampler (Fig. A1.1) is designed to obtain quantitative samples of macroinvertebrates from sediments in lakes, rivers, estuaries, oceans, and similar habitats.

TABLE A1.4 Advantages and Disadvantages of Commonly Used Core Samplers

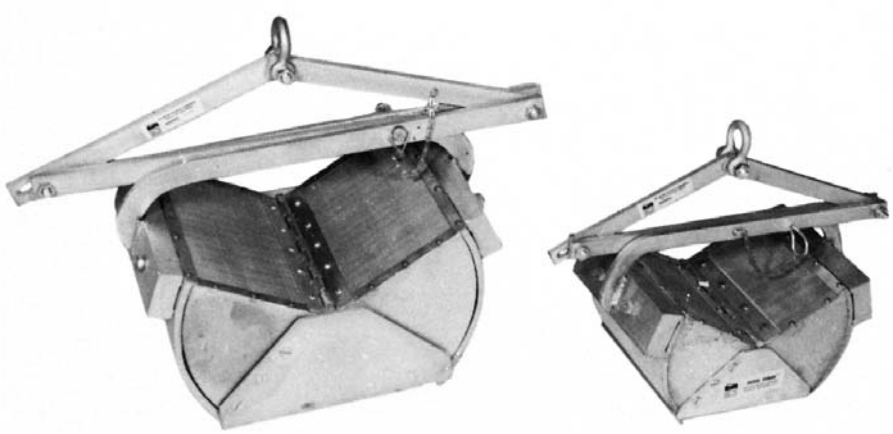
NOTE 1—Modified from Klemm et al., 1990 (31); Environment Canada, 1994(2); PSEP, 1997a(34) ; WDE, 1995(29); USEPA/ACE, 1998 (35)); USEPA (2001) (1).

Device/ Dimensions	Use	Depth Sample	Volume Sample, L^3	Advantages	Disadvantages
Fluorocarbon plastic or glass tube (3.5 to 7.5 cm inner diameter (I.D.); ≤120 cm long)	Shallow wadeable waters or deep waters if SCUBA available; soft or semi-consolidated deposits	0 to 10cm	0.096 to 0.44	Preserves layering and permits historical study of sediment deposition Minimal risk of contamination Rapid; samples immediately ready for laboratory shipment	Small sample size necessitates repetitive sampling
Hand corer with removable fluorocarbon plastic or glass liners (3.5 to 7.5 cm I.D.; ≤120 cm long	Same as above except more consolidated sediments can be obtained	0 to 10 cm	0.96 to 0.44	Same advantages as fluorocarbon plastic or glass tube Penetrates substrate with greater ease through use of handles	Small sample size necessitates repetitive sampling Requires careful handling to prevent spillage Requires removal of liners before repetitive sampling Barrel and core cutter metal may contaminate sample
Box corer	Same as above but the depth of the uncon-solidated sediment must be at least 1 m	0 to 70cm	≤ 30.0	Collects large, undisturbed sample; optimal for obtaining intact subsamples	Difficult to handle Relatively heavy; requiring larger vessel and power winch to deploy.
Gravity Corer, Phleger Corer (3.5 cm I.D., ≤50 cm long)	Deep lakes and rivers; semi-consolidated sediments	0 to 50cm	≤ 0.48	Reduces risk of sample contamination Maintains sediment integrity relatively well Penetrates with sharp cutting edge	Requires careful handling to avoid sediment spillage Requires repetitive and time-consuming operation and removal of liners due to small sample size
Gravity Corer, Kajak-Brinkhurst Corer (5 cm I.D., ≤70 cm long)	Deep lakes and rivers; Soft fine-grained sediments	0 to 70cm	≤ 1.37	Collects greater volume than the Phleger Corer.	Same as Phleger Corer
Benthos Gravity Corer (6.6, 7.1 cm I.D. <3 m long)	Soft, fine-grained sediments	0 to 3 m	≤ 10.26	Retains complete sample from tube because the core valve is fitted to the core liner Fins promote vertical penetration	Requires weights for deep penetration so the required lifting capacity is 750 to 1000 kg Requires vertical penetration Compacts sediment sample
Alpine Gravity Corer (3.5 cm I.D.)	Soft, fine-grained, semi-consolidated substrates	≤ 2 m	≤ 1.92	Allows different penetration depths due to interchangeable steel barrel	Lacks stabilizing fins for vertical penetration May penetrate non-vertically and incompletely Requires a lifting capacity of 2000 kg Disturbs sediment stratas and integrity Compacts sediment sample
Piston Corers	Ocean floor and large deep lakes; Most substrates	3 to 20 m	5 to 40	Typically recovers a relatively undisturbed sediment core in deep waters	Requires lifting capacity of > 2000 kg Piston and piston positioning at penetration may fail Disturbs surface (0 to 0.5 m) layer
BMH-53 Piston Corer	Waters < 2 m deep with extension rod; soft deposits	≤ 2 m	≤ 2	Piston provides for greater sample retention	Cores must be extruded onsite to other containers Metal barrels introduce risk of metal contamination
Boomerang Corer (6.7 cm I.D.)	Ocean floor (up to 9000 m deep)	1 m	3.52	Requries minimal shipboard equipment so small vessels can be used	Only penetrates 1.2 m Requires calm water for recovery Loses 10 to 20 % of sample
Vibracorer (5.0 to 7.5 cm I.D.)	Continental shelf of oceans, large lakes; sand, silty sand, gravelly sand substrates	3 to 6 m	5.89 to 13.25	For deep profiles it effectively samples most substrates with minimum disturbance Can be used in over 20 m of water depth Portablemodels can be operated from small vessels (e.g. 10 m long)	Labor intensive Assembly and disassembly might require divers Disturbs surface (0 to 0.5 m) layer Special generator may be needed Heavier models require larger boat and power winch to deploy

This device is most useful for collecting benthic macroinvertebrates from a wide range of bottom substrate types, for example, coarse sand, fine gravel, clay, mud, marl, and similar substrates. The sampler can be used in swift currents and deeper waters. The sampler is available in a range of sizes from 23 to 15 cm.



(*a*)



(*b*) (*c*)

FIG. A1.1 Ponar Grabs. (*a*) Screen-Top Sediment Grab, Standard Design (Photograph courtesy of Kahl Scientific Instrument Corp.); (*b*) Screen-Top Wildco Ponar Grab, Standard Design; (*c*) Wildco Petite Ponar Grab (Photograph courtesy of Wildlife Supply Co.)

A1.5.1.2 The Ponar grab sampler has paired jaws that should penetrate beneath the surface of the substrate without disturbing the water surface boundary layer of the substrate, close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. The Ponar collects a sample from an area of about 523 cm^2. A small version, the petite Ponar grab, takes a sample area of 232 cm^2 and can be used in habitats where there may be an unusual abundance of macroinvertebrates, thus eliminating the need to subsample. The Ponar grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

A1.5.1.3 *Hazards:*

(1) This device cannot be used in fast flowing streams, and in habitats with large cobble or rubble stone substrates.

(2) When not in use, a safety pin lock attached to the lever bar prevents closing of the sampler until the pin is removed.

(3) The weight of the Ponar grab makes it necessary to use a winch and cable or portable crane for retrieving the sample, and ideally the samples should be taken from a stationary boat or platform.

(4) The smaller version, petite Ponar grab, is designed for hand-line operation; however, the petite Ponar grab is enhanced by the use of a winch and cable.

A1.5.1.4 *Procedure:*

(1) Attach the Ponar grab to the cable and remove the safety pin with enough tension between the grab and cable so that the grip mechanism will release only when the sampler is on the bottom.

(2) The device should have a controlled lowering speed and should be lowered slowly because free-fall may airplane the device, causing the device to land improperly or causing a pressure wave and blowout of the surface layer of sediment when the grab reaches the bottom.

(3) Once the grab reaches the bottom, its weight will cause it to penetrate the substrate, and the slack-off on the cable allows the locking lever to release, therefore, permitting the movement that allows the horizontal locking bar to drop out of the locking notch and allows the jaws to close as the device is raised.

(4) Now the tension on the cable is resumed. As the grab is raised slowly, the lever system closes the jaws.

(5) Raise the sampler at a slow but steady rate to limit sample loss or washout.

(6) Once on board, empty into either a suitable container or a sieving device directly for processing.

(7) Thoroughly wash or hose the device with water so that all sediment material is included in the sample processing before a replicate sample is taken (see 10.4).

(8) Auxiliary jaw weight can be attached to the Ponar grab to increase its weight and is recommended for penetrating certain hard substrates.

A1.5.2 *Ekman Grab Sampler:*

A1.5.2.1 Ekman Grab Sampler (Fig. A1.2) is designed to obtain samples of macroinvertebrates from soft sediments in lakes, estuaries, oceans, and similar habitats where there is little current. This device is most useful for collecting macroinvertebrates from soft sediments, such as very fine sand, mud, and sludge. The sampler is available in sizes of 15 cm, 23 cm, and 30 cm.

A1.5.2.2 The Ekman grab sampler is a box-shaped device with two scoop-like jaws that should penetrate the intended substrate without disturbing the water surface boundary of the substrate, close when positioned properly on the bottom, and retain a discrete sample of sediment while it is brought to the surface for processing. Each half of the grab is covered with hinged doors to limit washout upon sample lowering and retrieval. The Ekman grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

A1.5.2.3 *Hazards:*

(1) This sampler is inefficient in deep waters, under adverse weather conditions, and in waters of moderate to strong currents or wave action.

(2) Exercise caution at all times once the grab is loaded or cocked because a safety lock is not part of the design.

(3) Operate the sampler from a boat with a winch and cable.

A1.5.2.4 *Procedure:*

(1) The sampler is cocked by raising each jaw upward into the cocked position using the attached cable and securing the cable to the catch pin located at the top of the sampler.

(2) Once cocked, lift the sampler overboard and lower slowly but steadily to the bottom.

(3) Once on the bottom, indicated by a slack line, the messenger is sent down the line tripping the catch mechanism, causing the spring loaded jaws to close the bottom of the sampler, containing the sediment.

(4) Raise the sample at a slow but steady rate to limit sample loss or washout.

(5) Once the sample is on board, empty the sample into either a suitable container or a sieving device directly for processing.

(6) Thoroughly wash or hose the device with water so that the entire sample is processed before a replicate sample is taken (see 10.4).

A1.5.3 *Petersen Grab Sampler:*

A1.5.3.1 The Petersen Grab Sampler is designed to obtain quantitative samples of macroinvertebrates from sediments in lakes, reservoirs, and similar habitats and is adaptable to rivers, estuaries, and oceans. This device (Fig. A1.3) is useful for sampling sand, gravel, marl, and clay in swift currents and deep waters. This sampler is available in a range of sizes that will sample an area from 0.06 to 0.099 m^2.

A1.5.3.2 The Petersen grab sampler has paired jaws that should penetrate the intended substrate without disturbing the water surface boundary layer of the substrate, close when positioned properly on the bottom, and retain the sample of sediment while it is brought to the surface for processing. The Petersen grab has been modified to improve its efficiency and reliability. Modified versions of the Petersen grab sampler may have a screened window at the top of each jaw to allow water to escape while the grab is descending and closing. While some modifications may close or function better, the sampling characteristics remain the same. Most of the modified versions are intended for use in estuarine and marine waters. A small version can be hauled aboard by hand and held with one hand for washing procedures.

A1.5.3.3 This grab sampler has limited application, and is not recommended for quantitative benthic work. A consensus of aquatic biologists consider the use of this device the least preferable grab sampler and would use it only in limited applications. The grab should only be used with consideration of its defects when quantitative estimates are attempted.

A1.5.3.4 *Hazards:*

(1) This grab sampler cannot be used under adverse weather conditions.

(2) It is advisable to use a winch and cable to lower and raise the sampler.

(3) Ideally a stationary boat or platform should be used when taking samples.

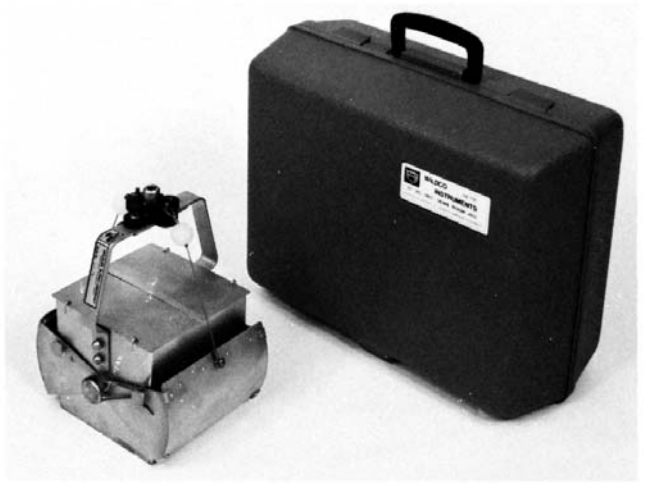
(4) Auxiliary weights can be added to each jaw to increase its weight for penetrating certain hard substrates.

(5) The modified Petersen devices are designed to be quite heavy and require heavy gear and a large vessel for efficient.

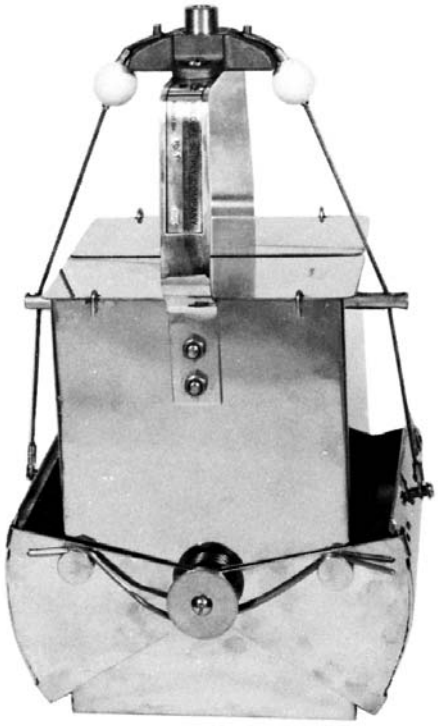
A1.5.3.5 *Procedure:*

(1) The Petersen grab sampler should be inspected for mechanical defects prior to use.

(2) The sampler is slowly lowered to the bottom when open to avoid disturbing lighter materials of the substrate.



(*a*)



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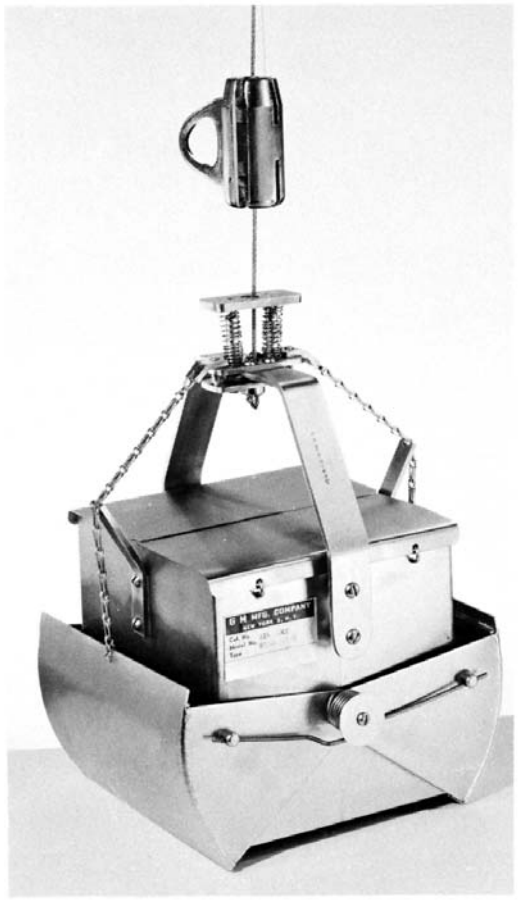
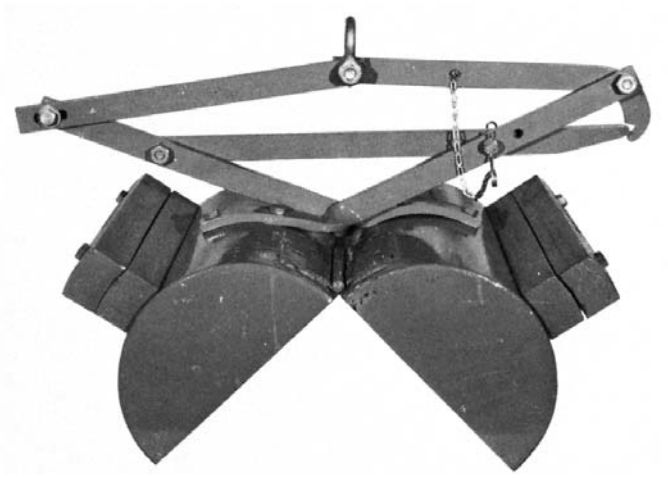


(*c*)

FIG. A1.2 Ekman Grabs. (*a*) Wildco Ekman Grab, Standard Design with Case; (*b*) Wildco Ekman Grab, tall design, (Photographs courtesy of Wildlife Supply Co.; (*c*) Ekman Box Sediment Grab (Birge-Ekman Design), (Photograph courtesy of Kahl Scientific Instrument Corp.)



(*a*)

(*b*)

FIG. A1.3 Petersen Grabs (*a*) Wildco Petersen Grab (Photograph courtesy of Wildlife Supply Co.); (*b*) Kahl Petersen Grab (Photograph courtesy of Kahl Scientific Instrument Corp.)

(3) When the lowering line is slackened, a catch is released, the two scoops close, and a semicircular bite of the sediment is taken. Raise the sampler at a slow but steady rate to limit sample loss or washout.

(4) Once the grab is aboard the vessel, empty the sample either into a suitable container or a sieving device directly for processing.

(5) Thoroughly wash or hose the device with water, so that all the sample is included in the sample processing before a replicate sample is taken (see 10.4).

A1.5.4 *Smith-McIntyre Grab Sampler:*

A1.5.4.1 Smith-McIntyre Grab Sampler (Fig. A1.4) is designed to obtain quantitative samples of macroinvertebrates from sediments in rough weather in hard sand bottoms in lakes, streams, estuaries, and oceans. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates and is useful under adverse weather conditions. This device samples a surface area of 0.1 m^2.

A1.5.4.2 The Smith-McIntyre grab sampler has paired jaws that are forced to penetrate into the intended substrate by two "loaded" springs, need to close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. The Smith-McIntyre grab sampler is fitted with gauze panels or free-swinging panels on the top to reduce the shock wave during descent. Larger Smith-McIntyre grabs can be constructed depending on the type of bottom to be sampled and additional weights can be fitted to the frame of the grab sampler for additional penetration into the sediment.

A1.5.4.3 *Hazards:*

(1) The spring-loaded jaws of the Smith-McIntyre grab should be considered a hazard and caution should be exercised when using the device.

(2) Due to the weight and size, this device should be used from a vessel with boom and lifting capabilities.

(3) Do not handle this device in the loaded mode except just prior to sampling.

A1.5.4.4 *Procedure:*

(1) The Smith-McIntyre grab is "loaded" by compressing the large coil springs mounted on the instrument using the loading bar.

(2) As soon as the spring is loaded, insert the safety pins to prevent the accidental triggering of the bottom plates.

(3) Once the device is overboard, just prior to being lowered to the bottom, remove the safety pins.

(4) Exercise caution to stand clear of the cocked jaws.

(5) The Smith-McIntyre is lowered slowly but at a steady rate by cable until the trigger plates contact the bottom.

(6) Pressure on these plates releases the two coiled springs that drive the buckets (jaws) into the sediment.

(7) Applying tension to the lifting cable completes the closure of the jaws, and the sampler may then be returned to the surface.

(8) Closure of the sampler is made at the side, rather than at the bottom.

(9) After closure the sample is given optimum protection from washout during return trip by the cylindrical configuration of the sampler.

(10) This device may be fitted with a hydraulic closure device that facilitates sampling in hard-packed bottoms, such as clay.

(11) Once on deck, place the sampler on a stand; the sample buckets can be disengaged from the rest of the device by releasing two retaining latches at each end of the upper semicylinder, and the sample is dumped into a large basin or washtub and prepared for processing.

(12) Thoroughly wash or hose the grab buckets with water so that all the sediment material is included in the sample processing before a replicate sample is taken (see 10.4).

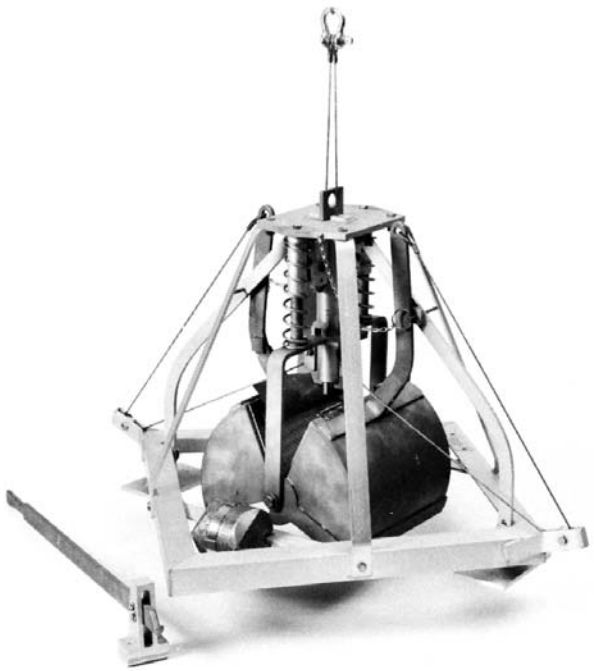


FIG. A1.4 Smith-McIntyre Grab (Photograph courtesy of Kahl Scientific Instrument Corp.)

(13) After the sample has been removed, the springs may then be loaded and the safety pins installed.

A1.5.5 *Van Veen Grab Sampler:*

A1.5.5.1 Van Veen Grab Sampler (Fig. A1.5) is designed to give quantitative samples of macroinvertebrates from sediments in estuaries, oceans, and similar habitats, and is adaptable to freshwater areas including large rivers. This device is useful for sampling sand, gravel, mud, clay and similar substrates. This sampler is available in two sizes, 0.1 m^2 and 0.2 m^2.

A1.5.5.2 The Van Veen grab sampler has paired jaws that should penetrate the intended substrate without disturbing the water surface boundary of the substrate, close by pincer-like action of two long arms when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. The Van Veen is basically an improved version of the Petersen grab in that long arms have been attached to the jaws to stabilize the grab on the bottom in the open sea just prior or during closing of the device. Additional weights can be applied to the jaws to effect greater penetration in sediments. The long arms give added leverage for penetrating hard sediments. Larger versions of this grab can be constructed depending upon the type of bottom to be sampled, and the type of vessel available to deploy this sampler. The Van Veen grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

A1.5.5.3 *Hazards:*

(1) At great ocean depths the sampler is sometimes difficult to operate as standing waves or swell at the surface or deeper down will act upon the levers so as to close the grab long before it reaches the bottom sediment.

(2) As with the larger grabs, the Van Veen should be lowered from a stationary vessel or platform with boom and lifting capabilities.

A1.5.5.4 *Procedure:*

(1) The Van Veen is cocked with the long arms assuming the spread condition.

(2) The chains from the jaws are attached to the counter balance mechanism, as are the slackened wires from the long arms.

(3) Tension is carefully applied to the triggering mechanism as the sampler is winched off its platform, and once the tension is firmly changed from the jaws, the Van Veen is relatively stable in the cocked position.

(4) Exercise care in lowering the Van Veen through the surface of the water as occasionally contact will produce slack in the chain that will trip the counter balance mechanism.

(5) The grab is lowered slowly to the bottom, and once it makes contact with the bottom the grab should be winched in, which initially closes the device and then raises it from the sediment.

(6) The grab is retrieved slowly to limit washout and once aboard the vessel, empty the grab into either a suitable container or a sieving device directly for processing.

(7) Thoroughly wash or hose the device with water so that all the sample is processed before a replicate sample is taken (see 10.4).

A1.5.6 *Orange-Peel Grab Sampler:*

A1.5.6.1 Orange-Peel Grab Sampler (Fig. A1.6) is designed to obtain quantitative samples of macroinvertebrates from sediments in marine waters and deep lakes. This device is useful for sampling sand, cobble, rubble stone, and similar substrates. The sampler is available in a range of sizes but the 1600 cm^3 is generally used although larger sizes are available. The sampler should not be used in critical quantitative work that is to be compared with results of other areas and is recommended as a reconnaissance sampler only.

A1.5.6.2 The Orange-Peel grab sampler has four curved jaws that close to encircle a hemisphere of sediment and should penetrate the intended substrate without disturbing the water

FIG. A1.5 Van Veen Grab (Photograph courtesy of Kahl Scientific Instrument Corp.)

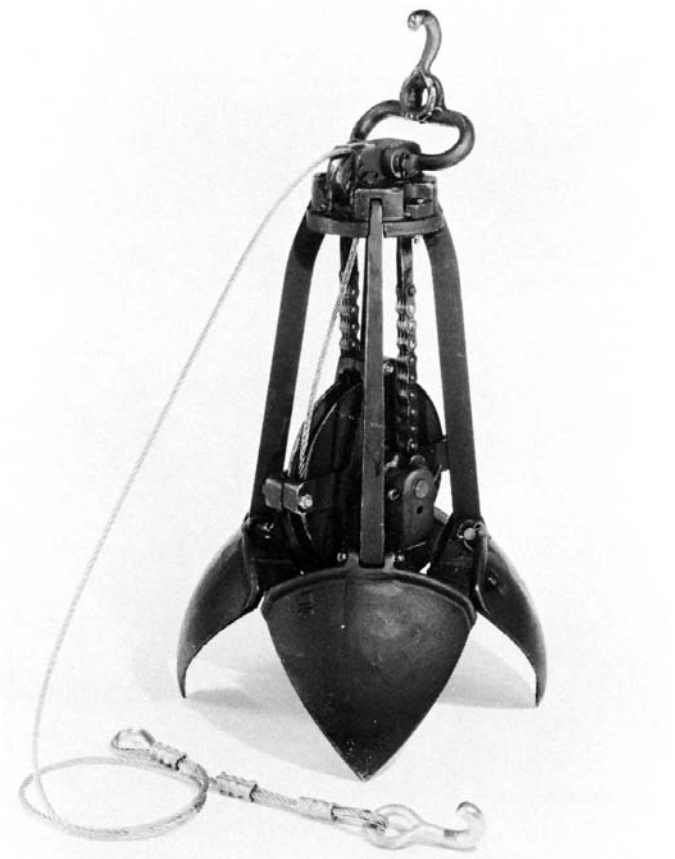


FIG. A1.6 Orange-Peel Grab (Photograph courtesy of Kahl Scientific Instrument Corp.)

surface boundary of the substrate, close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. A modification of the Orange Peel, described by Reish (1959 (**348**)) has a trigger mechanism and more efficient closing jaws, and the volume of sample to surface-area sampled relationship has been worked out. The surface area of this device also varies with penetration depth or volume sampled. The device penetrates to a maximum depth of 18 cm, but this depth will vary.

A1.5.6.3 *Hazards:*

(1) This sampler cannot be used under adverse weather conditions.

(2) The Orange Peel should be inspected for mechanical defects prior to use.

(3) When taking samples, a stationary boat or platform should be used.

A1.5.6.4 *Procedure:*

(1) Lower the sampler to the bottom by a powered winch and cable.

(2) Lower the sampler at a slow but steady rate.

(3) Once the sampler reaches the bottom, the jaws are operated by a large wheel and sprocket mechanism within the upper framework, and may be operated by a second cable or by a slack release mechanism activated by a messenger.

(4) The sampler is retrieved slowly, but to limit sample loss a loosely fitted canvas sleeve can be placed on the upper works to limit washing out of the sample.

(5) Once the sample is on board, empty it either into a suitable container or a sieving device directly for processing.

(6) Thoroughly wash or hose the device with water, so that all sediment material is included in the sample processing before a replicate sample is taken (see 10.4).

A1.5.7 *Okean 50 Grab Sampler:*

A1.5.7.1 Okean 50 Grab Sampler (See Holme, 1971 for illustration (**328**)) is designed to obtain quantitative samples of sediment and macroinvertebrates primarily in marine, estuarine, and large river habitats. This device is useful for collecting macroinvertebrates from sand, gravel, mud, clay, and similar substrates. The sampler is available in various sizes, generally a sampling area of 0.25 m^2. The Okean 50 grab sampler is used to collect qualitative and quantitative samples from different aquatic habitats containing benthic macroinvertebrates living on or in various types of substrates.

A1.5.7.2 The Okean 50 grab sampler has paired jaws that should penetrate the intended substrate without disturbing the water surface boundary of the substrate, close when positioned properly on the bottom, and retain discrete samples of sediment while it is brought to the surface for processing. This device is modified from the Petersen grab by the addition of a counter weight to release the twin jaws and the installation of opening lids in the top of the jaws so that water can flow through as the device is being lowered. The Okean 50 grab sampler retains

many of the disadvantages of the Petersen grab but is better for sampling in deep water.

A1.5.7.3 *Hazards:*

(1) The top of the sampler also contains hinged doors that are held open so that water can flow through as the unit is being lowered and closes when the grab reaches the bottom.

(2) The sampler has a counter weight release mechanism to prevent tripping in mid-water.

(3) The sampler can be weighted up to 150 kg to improve penetration into the substrate.

A1.5.7.4 *Procedure:*

(1) Slowly and carefully lower the sampler, otherwise, disturbance of the sediment will occur.

(2) The sampler is heavy and requires a boat with a powered winch and cable.

(3) Raise the sampler at a slow but steady rate to limit sample loss or washout.

(4) Once the sample is on board, empty it into either a suitable container or a sieving device directly for processing.

(5) Wash or hose the sampler with water so that all the sample is removed from the device for processing before a replicate sample is taken (see 10.4).

A1.5.8 *Shipek (Scoop) Grab Sampler:*

A1.5.8.1 The Shipek (Scoop) Grab Sampler (Fig. A1.7) is designed to obtain quantitative samples of macroinvertebrates from sediments in marine waters and large inland bodies of water. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates. It is designed to take a sediment sample with a surface area of 20 cm^2 to about 10 cm deep at the center.

A1.5.8.2 The Shipek scoop type grab sampler consists of a semicylindrical scoop and should be positioned properly on the bottom to take a scoop and retain discrete samples of sediment through 180°. Unlike many other types of samplers, closure of the device is made at the side, rather than at the bottom.

A1.5.8.3 *Hazards:*

(1) This sampler cannot be used under adverse wind and wave conditions.

(2) The sampler requires a vessel with a winch and cable.

A1.5.8.4 *Procedure:*

(1) The sampler should be lowered on a near vertical line.

(2) The sampler is composed of two concentric half cylinders, the inner semicylinder is rotated at high torque by two helically wound external springs.

(3) Upon contact with the bottom, the two external springs are automatically released by the inertia of a self-contained weight upon a sear mechanism which trips the catch and the scoop rotates upward.

(4) At the end of its 180° travel, the sample bucket is stopped and held at the closed position by residual spring torque.

(5) After closure the sample is given optimum protection from washout during the return trip by the cylindrical configuration of the sampler.

(6) The scoop can be disengaged from the upper semicylinder by releasing the two retaining latches.

(7) Once the sample is taken, it is retrieved by a power winch and cable.

(8) Once on deck the sample bucket may be disengaged from the rest of the device by releasing two retaining latches at each end of the upper semicylinder.

(9) Empty the sample into either a suitable container or a sieving device directly for processing.

(10) Wash or hose the sampler with water so that all the sample is processed before a replicate sample is taken (see 10.4).

A1.5.9 *Holme (Scoop) Grab Sampler:*

A1.5.9.1 The Holme (Scoop) Grab Sampler (Fig. A1.8) is designed to obtain quantitative samples of sediment and macroinvertebrates primarily in marine and estuarine waters and large deep freshwater lakes. This device is useful for sampling macroinvertebrates from sand, gravel, mud, clay, and similar substrates. This sampler is designed to take a sediment sample with a surface area of 0.05 m^2 and approximately 15 cm deep at the center. The device comes with a single scoop or double scoops.

A1.5.9.2 The Holme (scoop) grab sampler has a semicylindrical scoop mounted on the bottom of a heavy frame and needs to be positioned properly on the bottom to take a scoop to retain discrete samples of sediment through 180°. The device penetrates to a depth of about 150 mm. The sampler may be modified to include double scoops each of 0.05 m^2 or larger.

A1.5.9.3 *Hazards:*

(1) This sampler cannot be used under adverse wind and wave conditions and resetting of the scoop is somewhat awkward.

A1.5.9.4 *Procedure:*

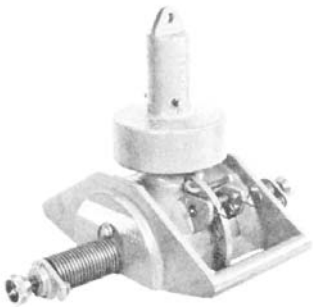


FIG. A1.7 Shipek (Scoop) Grab (Photograph courtesy of Hydro Products.)

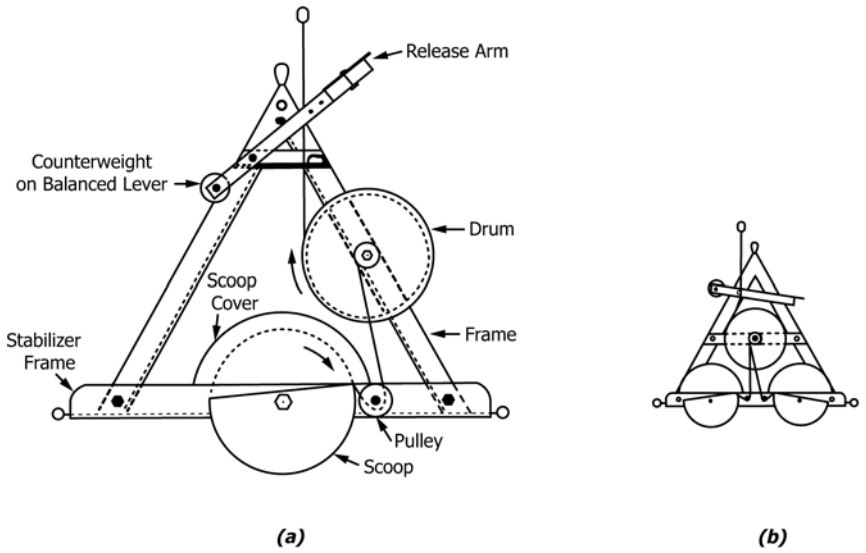


FIG. A1.8 Holme Grabs. (*a*) Single Holme; (*b*) Double Holme (See Holme and McIntyre (1971), pages 103–105)

(1) Slowly lower the sampler on a vertical plane with the scoop opening downward until it firmly contacts the substrate.

(2) The trip mechanism is released on lifting; the scoop forcibly rotates 180° along its horizontal axis.

(3) The sample is completely enclosed from below; a cover over the top limits washout.

(4) Operate the sampler from a boat with a powered winch and cable because of its bulk and weight.

(5) Once aboard the vessel, empty the sample into either a suitable container or a sieving device directly for processing.

(6) Thoroughly wash or hose the device with water, so that all the sample is included in the sample processing before a replicate sample is taken (see 10.4).

A1.5.10 *Surber Sampler:*

A1.5.10.1 The Surber sampler (Figs. A1.9 and A1.10) is designed to obtain a qualitative or quantitative sample of macroinvertebrates from a unit area. The device is used in shallow flowing streams and shallow areas of rivers with mud, sand, gravel, or rubble substrates. Modification of its basic design has resulted in other sampling devices, such as the portable invertebrate box sampler (Fig. A1.11). The latter closed-box-type sampler is preferred, if available. A variety of mesh sizes is available and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected. These devices sample an area of 0.1 m^2. The device is restricted to use in shallow streams or shallow areas of rivers, and it depends on a water velocity of not less than 0.05 m/s to wash the sample into a net.

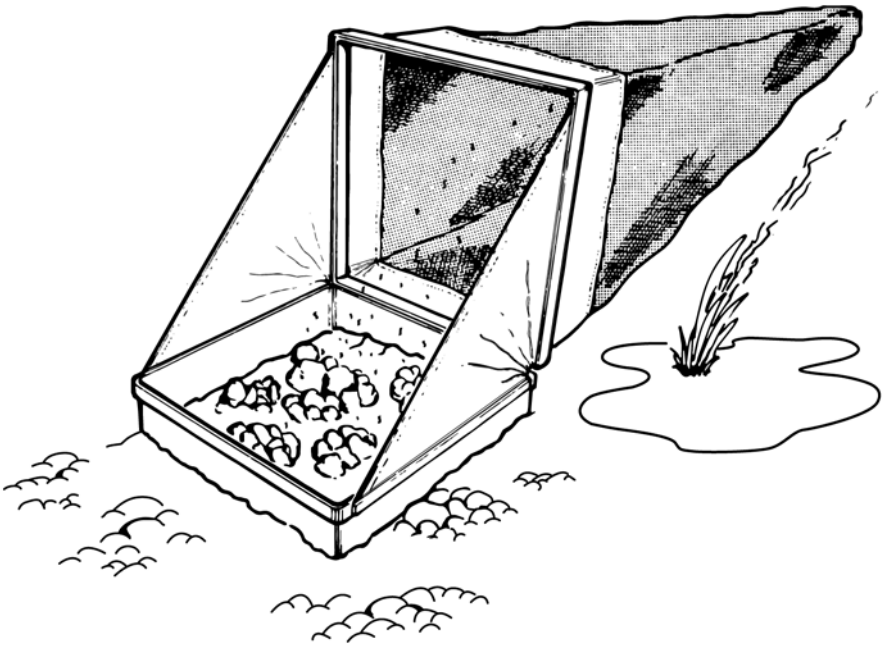


FIG. A1.9 Surber Sampler (Illustration courtesy of Kahl Scientific Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

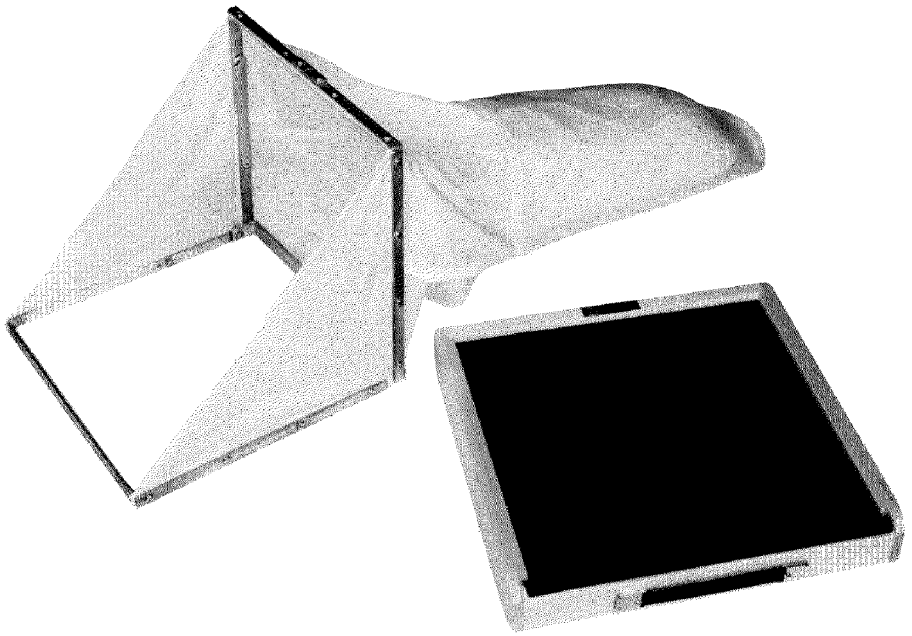


FIG. A1.10 Surber Sampler (Photograph courtesy of Wildlife Supply Co., 301 Cass St., Saginaw, MI 48602)

A1.5.10.2 The Surber sampler consists of two 30.5-cm frames, hinged together; one frame rests on the substrate, the other remains upright and holds the nylon net. The sampler is positioned with its net mouth open, facing upstream. When in use, the two frames are locked at right angles, one frame marking off the area of substrate to be sampled and the other frame supporting a net to strain out organisms washed into it from the sample area. Modification of the Surber sampler to overcome some of the limitations of its use (for example, loss of organisms due to backwash) has resulted in the design and construction of a number of related sampling devices, such as the four-sided (enclosed) portable invertebrate box sampler, the cylindrical Hess sampler, the cylindrical Hess stream bottom sampler, and the cylindrical stream-bed fauna sampler. Operation of the portable invertebrate box, Hess, Hess stream bottom, and stream-bed fauna samplers are similar to the Surber sampler.

A1.5.10.3 The Hess (cylindrical) sampler (Fig. A1.12) is designed to obtain a qualitative or quantitative sample of macroinvertebrates from a unit area. The device is used in shallow flowing streams and shallow areas of rivers with mud, sand, gravel, or rubble substrates. Modification of its basic design has resulted in other sampling devices, such as the Hess stream bottom sampler (Fig. A1.13) and stream-bed fauna sampler (Fig. A1.14). A variety of mesh sizes is available, and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected. The area sampled by these devices is dependent on their diameter and is comparable to the Surber sampler. These devices sample an area of 0.1 m^2.

A1.5.10.4 The net used to collect macroinvertebrates can vary in mesh size, length, taper, and material, for example, canvas, taffeta, or nylon monofilament. The net is usually made of nylon, and a variety of mesh sizes is available. The mesh size used will depend on the objectives of the study. A mesh size of 0.35 mm, for example, will retain most instars of aquatic insects. While a smaller mesh size might increase the number of smaller invertebrates and young instars collected, it will clog more easily and exert more resistance to the current than a larger mesh, possibly resulting in a loss of organisms due to backwashing from the sample net.

A1.5.10.5 It should be noted that these samplers are specific for macroinvertebrates, and that many of the microcomponents of the benthos will not be collected.

A1.5.10.6 The Surber, portable invertebrate box, Hess, Hess stream bottom, and stream-bed fauna samplers sample an area of 0.1 m^2.

A1.5.10.7 The polyester foam base of the portable invertebrate box sampler conforms to a variety of substrates to limit the loss of organisms from beneath the sampler. The Hess, Hess stream bottom, and stream-bed fauna samplers can be "turned" into most sediment types to a depth of several centimetres. The Surber sampler rests on the surface of most sediments.

A1.5.10.8 When sampling is completed, the net of the portable invertebrate box sampler slides out for cleaning or exchange with a different net. Hess-type samplers may have a mason jar ring and an adapter with a fixed or removable cloth net bucket.

A1.5.10.9 These samplers are designed for use in shallow, flowing waters. These samplers cannot be used as efficiently in still or deep water. These samplers are best used in water of 30.48-cm (1-ft) depth or less. If the water depth is greater than 30.48 cm (1 ft), benthic organisms may wash over the top of the net rather than into it. These samplers do not provide quantitative samples consistently, and the efficiency of the sampling device depends on the experience and ability of the user. While there can be large sampling errors associated with

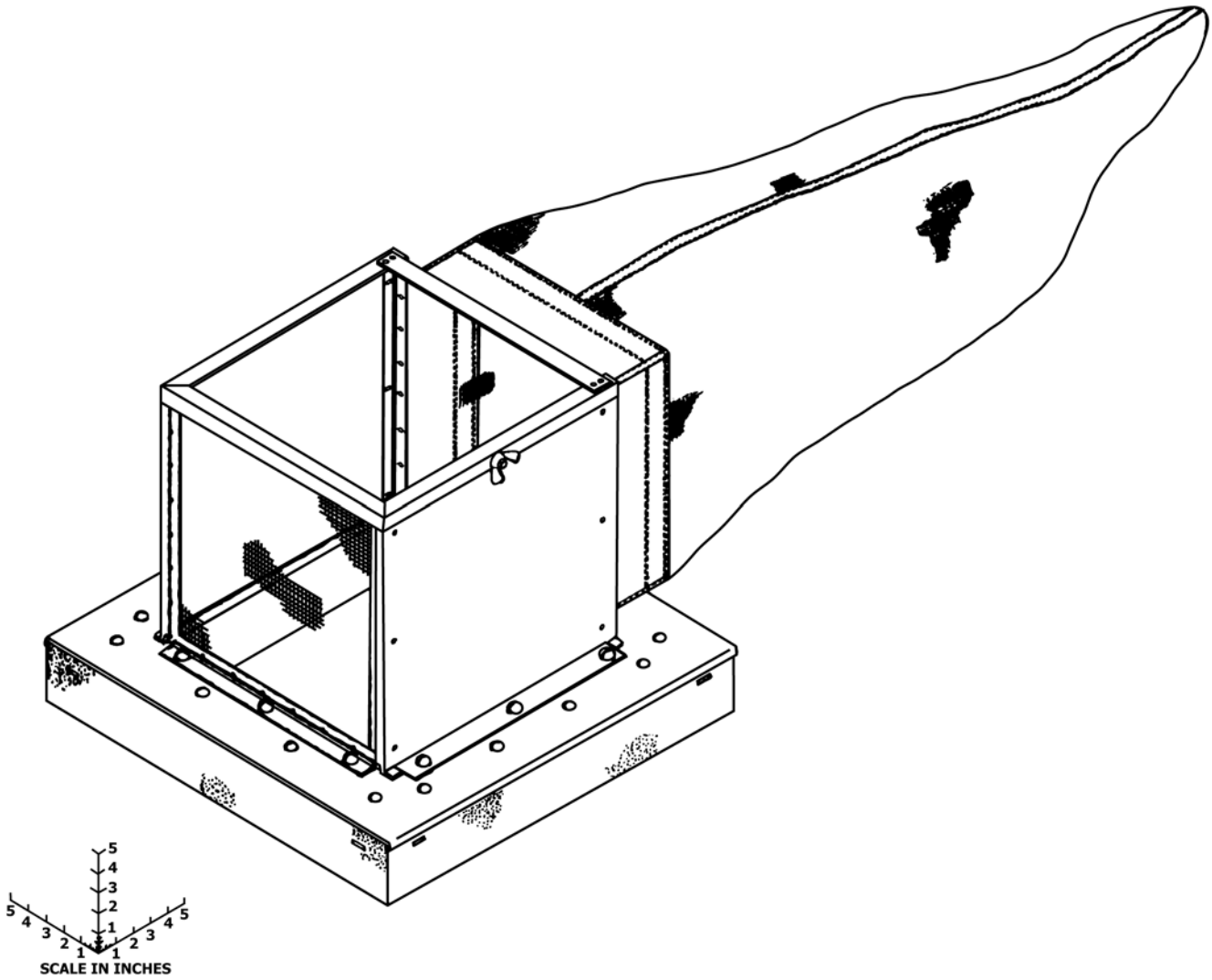


FIG. A1.11 Portable Invertebrate Box Sampler (Illustration courtesy of Ellis-Rutter Associates, P.O. Box 401, Punta Gorda, FL 33950)

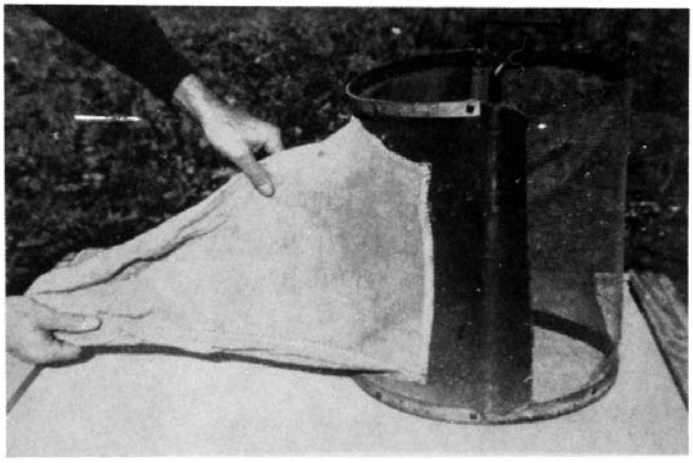


FIG. A1.12 Hess Sampler (Photograph courtesy of Billy G. Isom)

their use by an inexperienced operator, these samplers can provide data which are accurate and comparable if they are used consistently by one experienced person in similar habitats. If the water velocity is very great, resistance provided by

FIG. A1.13 Hess Stream Bottom Sampler (Photograph courtesy of Wildlife Supply Co., 301 Cass St., Saginaw, MI 48602)

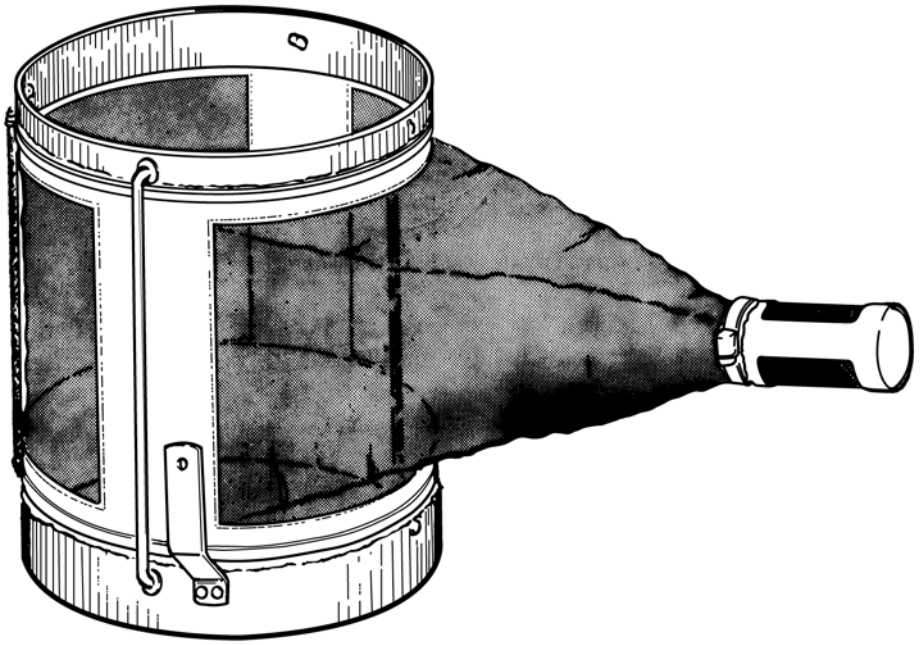


FIG. A1.14 Stream-Bed Fauna Sampler (Photograph courtesy of Kahl Scientific Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

the small mesh of the net or debris washed into it, or both, may result in a backwashing effect that washes benthic organisms out of the sample area of the Surber sampler or top of the other samplers.

A1.5.10.10 *Hazards:*

(1) Heavy gloves may be required when handling dangerous debris, for example, glass or other sharp objects present in the sediment.

A1.5.10.11 *Procedure:*

(1) Position these samplers securely on the substrate, parallel to the flow of the water, with the net pointing downstream.

(2) Bring the samplers down quickly to reduce the escape of rapidly moving organisms.

(3) There should be no gaps under the edges of the frame that would allow for washing of water under the net and loss of benthic organisms.

(4) Eliminate gaps that may occur along the edge of the Surber sampler frame by carefully shifting rocks and gravel along the outside edge of the sampler. This is also true of the cylindrical-type samplers if they are on rubble substrate that makes turning into the bottom difficult. The portable invertebrate box sampler polyester foam pad can conform to a relief of 7.6 cm (3 in.).

(5) Take care not to disturb the substrate upstream from the sampler, to avoid excessive drift into the sampler from outside the sample area.

(6) Once the sampler is positioned on the stream bottom, it should be maintained in position during sampling so that the area delineated remains constant.

(7) Hold the Surber sampler with one hand or brace with the knees from behind. The Hess, Hess stream bottom, and stream-bed fauna samplers, and the portable invertebrate box samplers can be held with one hand or braced with the knees from the sides. The portable invertebrate box sampler also can be sat upon for convenience while sampling; this provides the collector with a stable sampling platform that allows maximum manipulation of the substrate with little sampler movement.

(8) Turn over carefully all rocks and large stones and rub carefully in front of the net with the hands or a brush to dislodge the organisms clinging to them.

(9) Examine each stone carefully for attached or clinging organisms, larval or pupal cases, etc. before discarding.

(10) Scrape attached algae, insect cases, etc. from the stones into the sample net.

(11) Wash larger components of the substrate within the enclosure; water flowing through the sampler should carry dislodged organisms into the net.

(12) Stir the remaining gravel and sand vigorously with the hands to a depth of 10 cm (4.0 in.) where applicable, depending upon the substrate, to dislodge bottom-dwelling organisms.

(13) It may be necessary to hand pick some of the heavier mussels and snails that are not carried into the net by the current.

(14) If water level is too slow or low to allow continuous flow through the sampler, substrate can be hand-splashed into the net, although sampler efficiency will be reduced.

(15) Remove the sample by inverting the net (or washing out sample bucket, if applicable) into the sample container (wide mouthed jar).

(16) Examine the net carefully for small organisms clinging to the mesh, and remove them (preferably with forceps to avoid damage) for inclusion in the sample.

(17) Rinse the sampler net after each use (see 10.4).

A1.5.11 *Drift Net Samplers:*

A1.5.11.1 Drift net samplers (Figs. A1.15 and A1.16) are designed to obtain qualitative and quantitative samples of macroinvertebrates which drift in flowing streams and rivers with a velocity of not less than 0.05 m/s. Drift nets vary in size, but the type commonly used has an upstream opening of 15 by 30 cm, and the collection bag is 1.3 m long. A variety of mesh sizes is available, and mesh size should be selected based on the objectives of the study; the finer the mesh, the more organisms (instars) will be collected.

A1.5.11.2 Macroinvertebrate drift is a normal feature of flowing waters. Two functions are ascribed to drift: (*1*) distributes aquatic larvae over the whole stream and (*2*) provides a food supply for fish and invertebrates. Stress, fluctuations in water level, changes in light intensity, and changes in temperature are the basic factors that influence the extent of macroinvertebrate drift. Denuded and under populated areas of small streams and shallow rivers can be repopulated by numerous drifting organisms. These organisms may move an indefinite distance downstream where they again attach to the bottom substrate. A second source of drifting macroinvertebrates is the immature insects in the final stages of metamorphosis that actively seek to reach the water surface where emergence to the adult stage occurs. Regular periodic downstream drift rate of immature insects and other macroinvertebrate fauna in slow-moving streams or rivers is markedly reduced in comparison to lotic habitats with rapidly flowing water.

A1.5.11.3 Drift nets are useful for collecting macroinvertebrates that actively or passively enter the water column or that are dislodged from the substrate; naturally or by stress. They are particularly well-suited for synoptic surveys because they are light weight and easily transported. Thousands of organisms, including larvae of stoneflies, mayflies, caddisflies, and midges and other Diptera, may be collected in a sampling period of only a few hours. The drift net efficiently collects organisms originating from all types of substrates and a wide

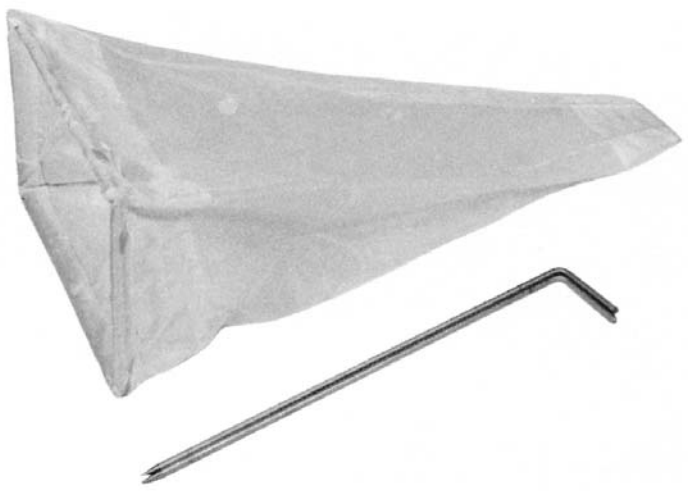


FIG. A1.15 Drift Net (Photograph courtesy of Wildlife Supply Co., 301 Cass St., Saginaw, MI 48602)

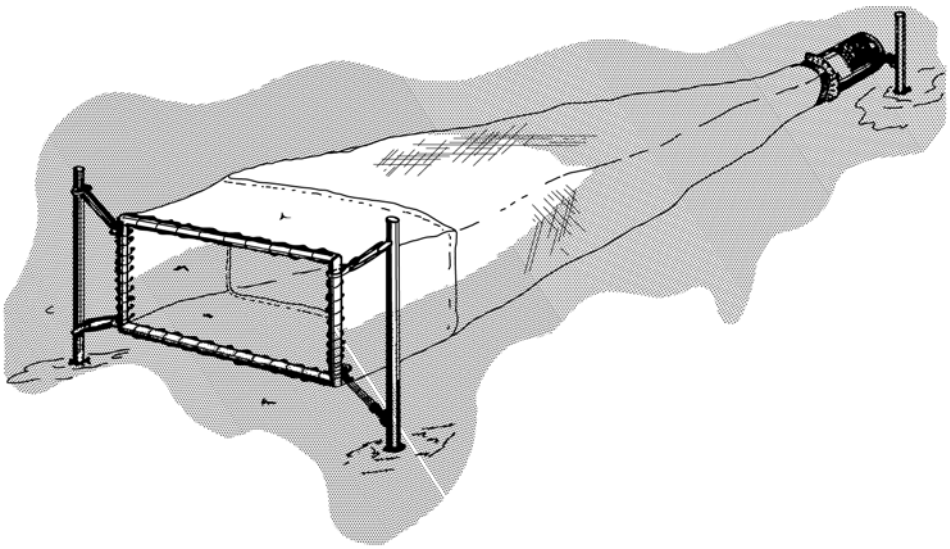


FIG. A1.16 Drift Net (Photograph courtesy of Kahl Instrument Corp., P.O. Box 1166, El Cajon, CA 92022-1166)

spectrum of microhabitats in lotic (flowing) waters. The device is restricted to flowing rivers or streams with a current velocity of more than 0.05 m/s.

A1.5.11.4 The typical drift net consists of a bag of nylon or nylon monofilament; a variety of mesh sizes can be used depending on the objectives of the study. The U.S. Standard No. 30 (0.595-mm mesh openings) net is often used for collecting macroinvertebrates. The frame typically consists of a 0.045-m^2 (15 by 30-cm) rod structure anchored into the stream bed by a pair of steel rods. Cable clamps are used to secure the nets to the rods.

A1.5.11.5 The average volume of water passing through the net is determined by measuring the water velocity at the mouth of the drift net with a current meter several times, and recording the total time the drift net is set in the water column. Several readings are taken, and the mean is used.

A1.5.11.6 The efficiency of the net is determined by the simultaneous measurement of the water velocity passing by the set drift net.

A1.5.11.7 The drift net frame can be fitted anteriorly with a mouth reducing rectangular plexiglass enclosure (Rutter and Ettinger, 1977 (**426**)) to increase filtration efficiency. The type of drift net and mesh size utilized will depend on the objectives of the study and the physical characteristics of the flowing water.

A1.5.11.8 Alternatives to the typical drift net include the waterwheel drift sampler (Pearson and Kramer,1969 (**427**)) which might be useful in large rivers or streams which can be reached by automobile. An automatic drift sampler (Muller, 1965 (**428**)) can be constructed that eliminates the need for an attendant at the sampling site during collection of as many as eight consecutive samples. A modified emergence-trap drift sampler (Mundie, 1964 (**429**); Cushing, 1964 (**430**)) is useful in streams with extremely high drift, where water is very turbid, or where a long sampling period is desired without clogging. The drift collection usually represents a wide spectrum of the habitats found in a stream.

A1.5.11.9 A benthic sample shows only what taxa were existing in the particular area (usually some fraction of a square meter, etc.) that was sampled. The great variation among benthic samples, even in a limited area, illustrates the necessity of several samples and the influence of selecting the collecting sites. One drift sample might be adequate for collecting the majority of invertebrate taxa in a stream reach, whereas a large number of benthic samples would be needed to cover the variety of bottom habitats even in a uniform reach of the stream. Quantitative benthic sampling is seldom extended to include stream banks, organic substrates (logs, etc.), and areas of dense vegetation. The drift net collects organisms from all these areas. Drift net collections often require much less sorting work than a series of benthic samples.

A1.5.11.10 Nets are light-weight and easy to set up in a stream and usually yield a light-weight sample. Benthic sampling in flowing water often procures samples heavy with inorganic materials. Drift samples of organic materials do not require the laborious, time-consuming job of washing out silts and clays and sorting and picking through much of the debris for the organisms in the samples.

A1.5.11.11 A drift net is inexpensive to construct, whereas bottom samplers are often costly and more than one kind may be required to adequately sample the multiple habitat types present in a stream or river.

A1.5.11.12 Drift collections can be used to determine drift density, rate, and periodicity of drift organisms, and interesting aspects of the organisms' life histories, for example, period of transformation.

A1.5.11.13 Drift collections often include terrestrial organisms that have fallen into the stream and which contribute to the food supplies of fish.

A1.5.11.14 Certain aquatic organisms enter the drift only sporadically and might be missed even though common in the benthos. The relative abundance of macroinvertebrates in a drift sample often differs significantly from their relative

abundance on the stream bottom. A slight current is necessary if a drift collection is to be taken (greater than 0.05 m/s).

A1.5.11.15 Most species and number of organisms drift more abundantly at night, so that the best collections are usually taken in the dark.

A1.5.11.16 There is a waiting period while the drifting organisms accumulate in the net.

A1.5.11.17 Tree leaves in the autumn, floating and anchor ice in the winter, and heavy debris (logs) during floods may interfere with drift net collecting and make processing difficult.

A1.5.11.18 The abundance and composition of drift changes daily, hourly, or seasonally and might prevent direct comparison of collections taken at different times. At times certain life stages of an organism might not be fairly represented in the drift.

A1.5.11.19 Drift collections give little precise habitat information for individual organisms, since the exact source of the individual is not known.

A1.5.11.20 Collections of drift, with the organisms originating an indefinite distance above the collecting site, may not show local or temporary deleterious effects imposed on an aquatic community, whereas bottom samples might reveal the destruction or reduction of benthos in a small area.

A1.5.11.21 *Hazards:*

(1) No specific hazards have been identified for use of drift nets.

A1.5.11.22 *Procedure:*

(1) Because the performance and sampling efficiency of a drift net sampler varies with local stream conditions, seasonal changes, and water level, make a preliminary test before the start of regular drift sampling in order to determine the best sampling stations, best sampling interval, number of nets needed, mesh size, and best sampling depth.

(2) For synoptic surveys, one net set above each of the major areas of population concentrations is usually adequate; but for definitive studies, locate stations so that drift can be evaluated from above a location of concern, from the location of concern, and below the area of concern.

(3) Take into consideration the fact that the drift net will collect drifting organisms that may have entered the drift from an indefinite distance upstream.

(4) Nets located 80 to 100 m below the location of concern will generally sample this location efficiently. A drift net below a riffle collects more animals than one below a pool.

(5) Drift insects are about evenly distributed at all levels in a stream, but in large rivers drift is more abundant near the bottom in the shoreline zone.

(6) It is generally found that there are pulses of drift organisms that move from top to bottom of the water column, at least during periods of low flow.

(7) For definitive studies, install two nets at each station-one about 25 cm from the bottom and one about 10 cm below the surface in water not exceeding 3 m in depth.

(8) If the objective of the study is to relate pupal exuviae to contamination, or to collect terrestrial organisms that may float on the surface, then extend slightly one net above the surface.

(9) Ideally, collect 24-h drift samples; but this is usually not practicable unless one resorts to the use of a water-wheel, automatic drift sampler, or a modified drift sampler with a restricted opening to solve the clogging problem.

(10) Although the sampling interval will vary with time of day, current velocity, density of drift organisms, and floating debris, collect 3-h daytime drift samples when either a 24-h or overnight sampling period is not prudent.

(11) Try to avoid using drift nets for large rivers with currents less than 0.05 m/s.

(12) Drift nets are anchored in the stream by driving ½-in. steel rods into the stream bottom or mounting the rods in concrete slabs that are weighted down with stones.

(13) Drift nets have also been used from small boats in large rivers (Rutter and Ettinger, 1977 (**426**)).

(14) Use cable clamps to secure the nets to the rods.

(15) Because the size of the catch varies as the flow of water through the net varies, it is necessary to measure the current velocity at the entrance of each net at the beginning and end of each sampling period so that the catch can be converted into number of organisms per volume of water flowing through the net.

(16) At the end of the specified sampling period, remove the net from the water by loosening the cable clamps and raising the net over the top of the steel rods, taking care not to disturb the bottom upstream of the net.

(17) Concentrate the material in the net in one corner by swishing up and down in the water and then wash into a bucket half-filled with water.

(18) Then sieve and handle the sample in the regular manner.

(19) Subdividing the sample substantially reduces analysis time with large samples (Waters, 1969b (**431**) and Weber (**300**)).

(20) Reporting data as numbers of individuals per net is meaningless because no two drift net samples are collected under exactly the same conditions of current velocity, stream discharge, and sampling interval.

(21) Conversion equations and other statistical aspects of drift sampling are given by Elliott, 1981 (**293**).

(22) An equation for converting the data to number per 100 m^3 of water flow is:

$$X = \frac{100a}{bdc} \qquad \text{(A1.1)}$$

where:

X = number of organisms per 100 m^3,
a = number of organisms in the net (density),
b = number of minutes of the sampling interval,
c = current velocity, m/min, and
d = area of the net opening (m^2).

(23) The first step in interpreting drift data is to determine the respective contributions of constant, behavioral, and catastrophic drift to the samples being analyzed.

(24) Only constant and behavioral drift are usually utilized in a synoptic survey, but catastrophic drift is extremely important in testing for recent discharges of toxic materials.

(25) Bear in mind that the drift density may not be a function of the total bottom population density or of production; however, species composition of the drift is useful as an index of species composition of the benthos.

(26) Density and composition of invertebrate drift are influenced by many factors that also should be considered when interpreting the data, including stage of life cycle, weather, time of day, light intensity, population density, temperature, turbidity, water level fluctuation, season, current velocity, growth rate, photoperiod, and proximity to tributary streams.

(27) In an enriched stream there is usually a marked increase in total numbers and biomass of drifting organisms as the stream becomes more polluted. Intolerant forms decrease and pollution tolerant forms increase proportional to changing water quality.

A1.5.12 *Basket Samplers:*

A1.5.12.1 Basket samplers are a highly effective device for evaluating the biological integrity of surface waters and for studying macroinvertebrate communities (**432-457**). The materials used in the basket sampler are natural or artificial materials of various compositions and configurations. The device is placed in water for a predetermined exposure period and depth for the colonization of macroinvertebrate communities. Basket samplers are used to collect qualitative and quantitative samples from lentic and lotic waters containing benthic macroinvertebrates living on various types of substrates. Physical factors such as stream velocity and depth may variably affect the degree of colonization. The sampling method is selective for drifting organisms (biased for insects) and for those that preferentially attach to or live on hard surfaces. Basket samplers are excellent for water quality monitoring; contain uniform substrate types at each station for better comparison; provide quantitatively comparable data; contain negligible amounts of debris, permitting quick laboratory processing; and usually do not require additional weight for stability. Basket samplers sample a known area at a known depth for a known exposure period. Basket samples provide no measure of the biota and condition of the natural substrate at a station. They record only biota accumulated during the exposure period.

A1.5.12.2 Basket samplers are usually colonized by a wide variety of macroinvertebrates that actively and passively enter the current or the water column. The use of basket samplers facilitates the consistent collection of samples. Consistent sampling is especially desirable when the results from different investigators and environments are to be compared.

A1.5.12.3 The basket sampler can be used alone or can effectively augment bottom substrate sampling, because many of the physical variables encountered in bottom sampling are minimized (for example, variable depth and light penetration, temperature differences, and substrate types).

A1.5.12.4 The type of basket sampler normally used (Fig. A1.17) is a cylindrical "barbecue" basket 11 in. (28 cm) long and 7 in. (17.8 cm) in diameter that is filled with approximately 17 lb (7.7 kg) of natural rocks varying from 1 to 3 in. (2.5 to 7.6 cm) in diameter (Mason 1967, 1971 (**447, 448**). A hinged door on the side provides access to the contents. An estimated 3.2 ft2 (0.3 m^2) of surface area is provided for colonization by macroinvertebrates. A ⅛-in. (3.2-mm) wire cable is passed through the long axis of the basket; one end is fastened with a cable clamp, and the other end is fixed to the float. A5-gal (19-L) metal container filled with polyurethane foam can be used as a float. A ⅜-in. (9.5-mm) steel rod threaded at each end is passed through the long axis of the float and fastened at each end by nuts. Three inch by 1⅛-in. by ⅛-in. (76.2 by 25.6 by 3.2-mm) strap iron serves as a swivel at each end, secured on the rods by nuts. The wire cable used to suspend the basket is attached to the swivels by holes drilled for that purpose. The float can be attached to a stationary structure, or the basket can be anchored to the bottom in shallow water.

A1.5.12.5 The rugged construction of the sampler is heavy enough to resist movement by water currents. Samples usually contain negligible amounts of extraneous material, permitting rapid laboratory processing.

A1.5.12.6 A collapsible type of basket sampler has been used for comparing populations surrounding rocky substrates (Bull, 1968 (**438**)). The sampler consists of a collapsible basket surrounded by a nylon netting bag that can be loaded with materials simulating the natural substrate on which it lies. A rim around the top helps retain the substrate material. When lowered to the bottom, the basket sampler collapses to form a substrate area that is eventually colonized. When the basket is raised off the bottom, the basket extends to its original hemispherical shape, and the surrounding net bag limits the loss of invertebrates during retrieval.

A1.5.12.7 *Hazards:*

(1) Samplers and floats may be difficult to anchor; they may be a navigation hazard.

(2) Samplers are susceptible to vandalism and often lost.

(3) Caution should be exercised in the reuse of samplers that may be subjected to contamination by chemicals.

A1.5.12.8 *Procedure:*

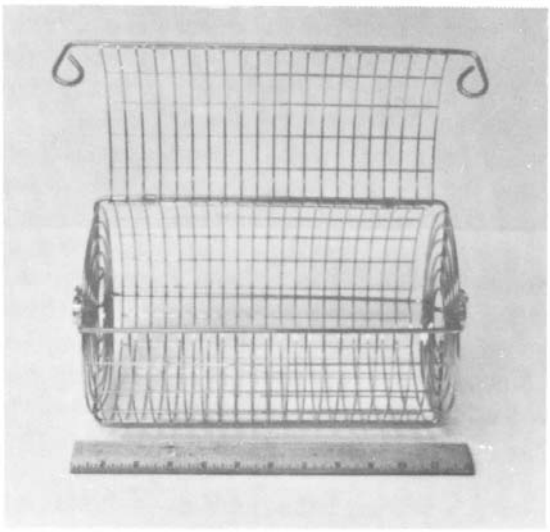
(1) In deep water, three basket samplers are suspended from floats, cement structures, or rods driven into the streambed or lake-bed and positioned well up in the euphotic zone of good light penetration (1 to 3 ft (0.3-0.9 m)) for maximum abundance and diversity of the macroinvertebrates. A4-ft (1.2-m) depth is acceptable unless the water is exceptionally turbid.

(2) The optimum period for substrate colonization is six weeks for most types of water. At least 3 replicate samples at each station should be evaluated.

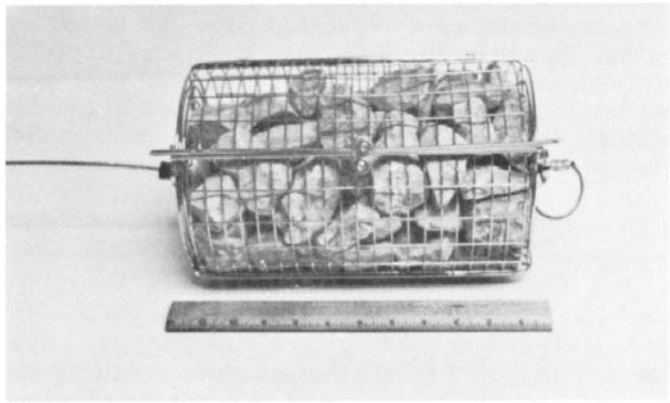
(3) For uniformity of depth, suspend the basket samplers from floats on ⅛-in. (3.2-mm) steel cable. If vandalism is a problem, use subsurface floats or put the samplers on supports placed on the bottom. Regardless of the installation technique, use uniform procedures (for example, the same depth and exposure period, sunlight, current velocity, and habitat type).

(4) At shallow water stations (less than 4 ft (1.2 m) deep), install the samplers so that the exposure occurs midway in the water column at low flow. The samplers may be installed in pools, runs, or riffles suspended below the water surface. The collections should be as representative of the reach as possible by ensuring that the samplers are not close to the bank.

(5) In streams up to a few meters in width, install the device at approximately midstream. In larger streams, install the device at approximately one quarter of the total width from the nearest bank.



(*a*)



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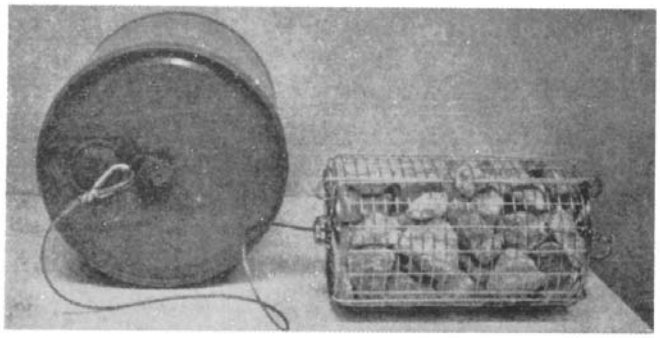

(*c*)

FIG. A1.17 Cylindrical "Barbecue" Basket Sampler: (A) Basket Sampler Empty; (B) Basket Sampler Containing Limestone Rocks and Ready for Installation; and (C) Basket Sampler Containing Limestone Rocks and Attached to 5-gal (19-L) Metal Container Filled with Polyurethane Foam. (Barbecue Baskets Available from Tenaco, 2007 NE, 27th Ave., Gainesville, FL 32609 or W.C. Bradly Enterprises, Inc., P.O. Box 1240, Columbus, GA 32993.)

(6) If the samplers are installed in July when the water depth is approximately 4 ft (1.2 m) and the August average low flow is 2 ft (0.6 m), the correct installation depth in July is 1 ft (0.3 m) above the bottom. The sampler will receive sunlight at optimum depth (1 ft (0.3 m)) and will not be exposed to air anytime during the sampling period. Care should be exercised not to allow the sampler to touch bottom, which may permit siltation, thereby increasing the sampling error.

(7) In shallow streams with sheet rock bottoms, basket samplers can be secured to ⅜-in. (0.95-cm) steel rods that are driven into the substrate or secured to rods that are mounted on low, flat, rectangular blocks half way between the water surface and the stream bed. However, these should be anchored securely to the rock bottom to avoid loss during floods.

(8) Factors such as the time of the year and the body of water sampled should be considered in the determination of exposure time. The exposure time should be consistent among sites during the study. If study time limitations reduce this period, the data should be evaluated with caution, and in no case should data be compared from samplers exposed for different time periods.

(9) Samplers should be protected from loss of invertebrates during retrieval. Most insects rapidly leave the sampler when disturbed; thus a retrieval method to limit their escape should be used.

(10) In shallow water, approach the basket samplers from downstream, lift the sampler quickly, and place the entire sampler in a polyethylene bag or jug containing the selected fixative. The fixative should be used only if the specimens collected require special processing for identification.

(11) Once the sampler is touched, it should be removed from the water immediately or many of the animals will leave the sampler. If the sampler has to be disturbed during the recovery process so that it cannot be lifted straight up out of the water, a net should be used to enclose the sampler before it is disturbed.

(12) To accomplish this, the rock-filled basket sampler should be enclosed either in a sieving bucket with U.S. Standard No. 30 sieve screen or by a dip net constructed of U.S. Standard No. 30 sieve or finer mesh bolting cloth that can be pulled around the sampling device before retrieval. Also, samplers exposed in deep water may be enclosed in a retrieval net and brought to the surface by divers. If the sampler can be pulled quickly from the water without undue disturbance, as described in 7.10, it may not be necessary to enclose it.

(13) The organisms can be removed in the field by disassembling the sampler in a tub or bucket partially filled with water and scrubbing the rocks with a soft-bristle brush to remove clinging organisms. The contents of the bucket are then poured through a No. 30 or 60 sieve and washed into a jar and preserved. If the organisms are not removed in the field, the basket samplers can be taken to the laboratory and disassembled if placed in a water-tight container containing a fixative or preservative. The samples should be labeled with at least the location, habitat, date, and time of collection.

(14) Cleaned basket samplers can be reused unless there is reason to believe that contamination has occurred. These substances may be toxic to the macroinvertebrates or may inhibit colonization. Do not reuse a basket sampler substrate that has been exposed to preservatives.

A1.5.13 *Multiplate Samplers:*

A1.5.13.1 Multiple-plate samplers consist of artificial substrate surfaces (tempered hardboard or ceramic plates) for colonization by aquatic organisms. Their uniform shape and texture compared to natural substrates simplifies the problem of sampling relative to basket samplers. Multiple-plate samplers are usually colonized by a wide variety of macroinvertebrates that actively and passively enter the current or the water column. The multiple-plate sampler can be used either alone or can effectively augment bottom substrate sampling because many of the physical variables encountered in bottom sampling are minimized (for example, variable depth and light penetration, temperature differences, and substrate types).

A1.5.13.2 The sampler can be purchased or constructed from readily available materials. Multiple-plate samples have been constructed of 8 or more tempered hardboard or ceramic material cut in 76 mm (3 in.) square or circular plates and separated by a specific arrangement of spacers. The plates and spacers are placed on a ¼-in. eyebolt. Total surface area of the 8-plate sampler is approximately 939 cm^2 (0.09 m^2), and the 14 plate sampler is 1160 cm^2 (0.116 m^2). The 14 plate, tempered hardboard, multiple-plate sampler weighs about 1 lb (0.45 kg).

A1.5.13.3 *Description of the Modified Hester-Dendy Multiple-Plate Sampler*—The modified multiple-plate (Fig. A1.18) is constructed of 0.25 in. (0.3 cm) tempered hardboard or ceramic material with 3 in. (7.6 cm) round or square plates and 1 in. (2.5 cm) round spacers that have ⅝-in. holes drilled in the center (Fuller, 1971 **(458)** and Cairns, 1982 **(459)**). The plates are separated by spacers on a 0.25-in. (0.63 cm) diameter eyebolt, held in place by a nut at the top and bottom. A total of 14 large plates and 24 spacers are used in each sampler. The top nine plates are each separated by a single spacer, plates 9 and 10 are separated by two spacers, plates 11 and 12 are separated by three spacers, and plates 13 and 14 are separated by four

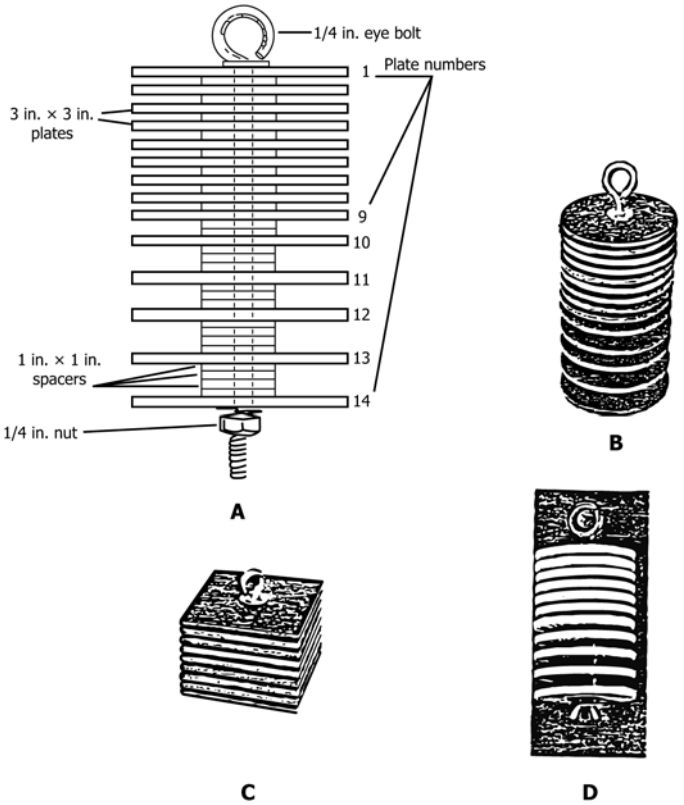

FIG. A1.18 Artificial multiple-plate samplers: (*a*) schematic drawing of multiple-plate sampler; (*b*) modified round; (*c*) original square, tempered hardboard, Hester-Dendy samplers; and (*d*) round ceramic multiple-plate macroinvertebrate sampler

spacers. The hardboard sampler is about 5.5-in. (14 cm) long, 3-in. (7.6 cm) diameter, exposes about 1160 cm^2 (0.116 m^2) of surface area for the attachment of organisms, and weighs about 1 lb (0.45 kg). The ceramic sampler is 6.5-in. long and weighs 2.2 lb (1 kg). The ceramic plates can be chemically cleaned, oven dried and reused indefinitely as they are stable and unaffected by long-term immersion in water. The sampler will not warp with time; therefore, the spacings between plates do not change, assuring replicate and efficient sampling. Each sampler is supplied with a 20-ft (6 m) long nylon suspension rope. The total weight is 2.2 lb (1 kg). Sturdy wire stakes for holding the sampler above the riverbed are recommended accessories.

A1.5.13.4 Another type of modified Hester-Dendy multiple-plate artificial substrate sampler (Ohio EPA, 1987 **(460)**) is constructed of ⅛-in. tempered hardboard cut into 3-in. (7.6 cm) square plates and 1-in. (2.5 cm) square spacers. A total of eight plates and twelve spacers are used for each sampler. The plates and spacers are placed on a ¼-in. eyebolt so that there are three single spaces, three double spaces, and one triple space between the plates. The total surface area of the sampler, excluding the eyebolt, is 145.6 $in.^2$ (939 cm^2 or 0.09 m^2). Five samplers are placed in streams tied to a concrete construction block which anchors them in place and prevents the multiple-plates from coming into contact with the natural substrates.

A1.5.13.5 The recommended exposure period for multiple-plate sampler is six weeks, and the time of exposure may be

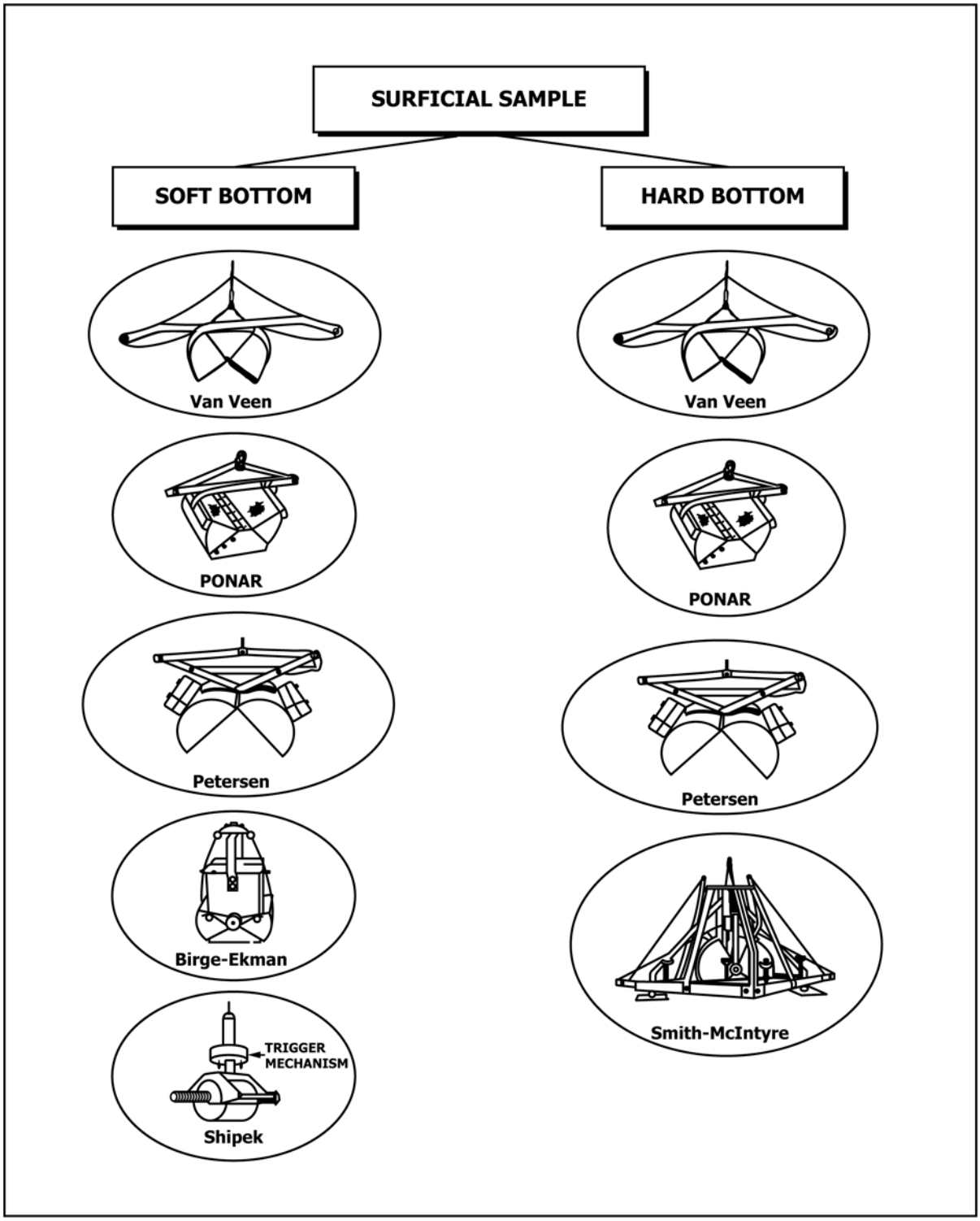

FIG. A1.19 Some Recommended Devices for Collecting Surficial Sediments (drawings from Murdoch and Azcue 1995 (46); USEPA 2001 (1))

critical to development of a relatively abundant and diverse community of organisms. Three replicate samples at each station are an absolute minimum. Collecting five replicate samples at each station will increase statistical precision and accuracy. Multiple-plate samplers are a highly effective device for evaluating the biological integrity of surface waters and for studying macroinvertebrate communities (**461-478**). Multiple-plate samplers are used to collect qualitative and quantitative samples from lentic and lotic waters containing benthic macroinvertebrates living on various types of substrates. Physical factors such as stream velocity and depth may variably affect degree of colonization. The sampling method is selective for

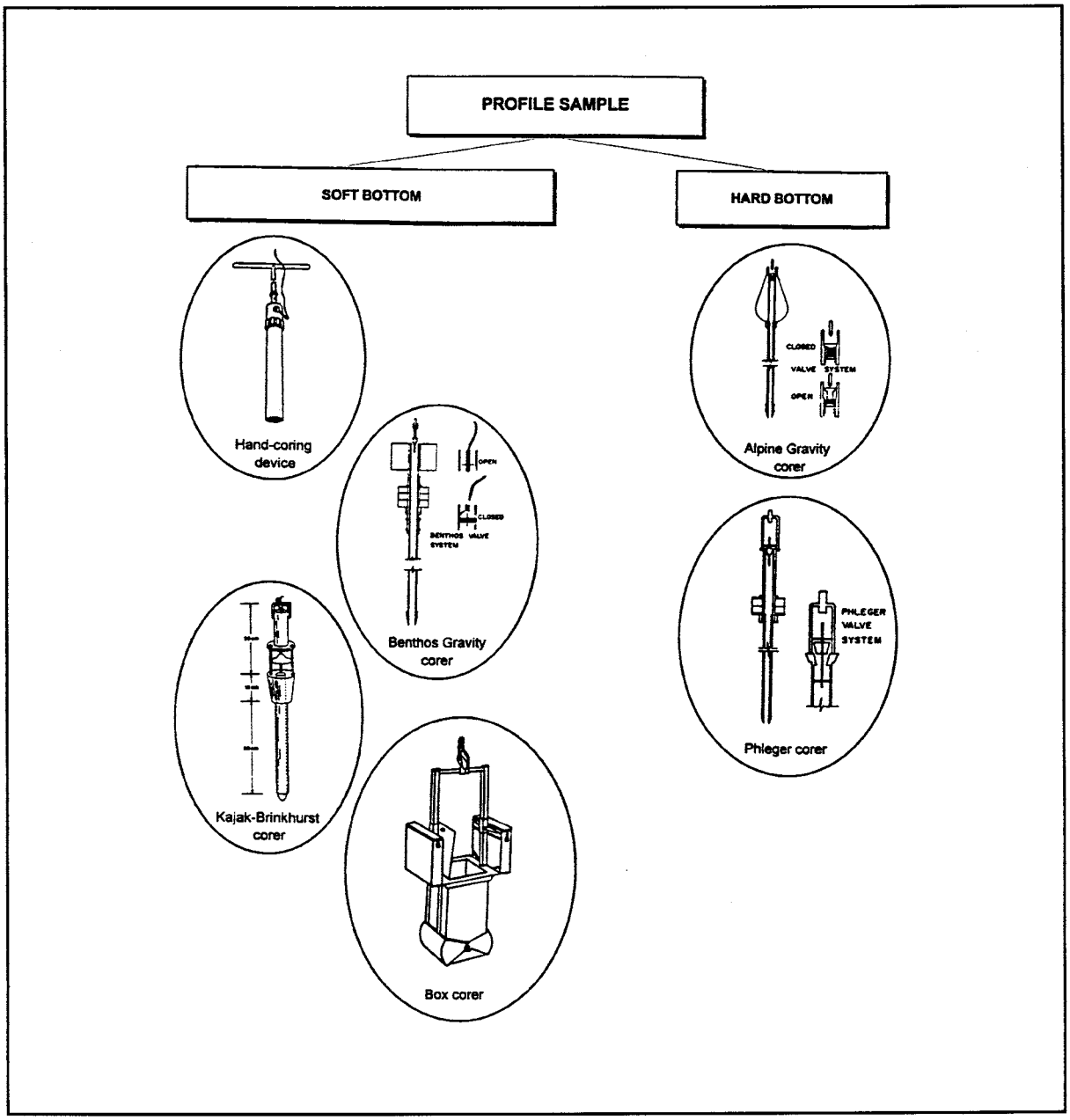

FIG. A1.20 Some Recommended Devices for Obtaining Sediment Profiles (drawings from Murdoch and Azcue 1995(46); USEPA 2001 (1))

drifting organisms (biased for insects) and for those which preferentially attach to or live on hard surfaces.

A1.5.13.6 Multiple-plate samplers are excellent for water quality monitoring, contain uniform substrate type at each station for better comparison, give quantitatively comparable data, contain negligible amounts of debris permitting quick laboratory processing, but may require additional weight for stability. Multiple-plate samplers sample a known area at a known depth for a known exposure period. Multiple-plate samples provide no measure of the biota and condition of the natural substrate at a station. They record only biota accumulated during exposure period. The distinct advantages of the multiple-plate sampler are its small size and light weight. It is the most adaptable of the recommended benthic invertebrate artificial substrate devices.

A1.5.13.7 *Hazards:*

(1) Samplers and floats may be difficult to anchor; they may be a navigation hazard.

(2) Samplers are susceptible to vandalism and often lost.

(3) Caution should be exercised in the reuse of samplers that may be subjected to contamination by chemicals.

A1.5.13.8 *Procedure:*

(1) In deep water three multiple-plate samplers are suspended from floats, cement structures, or rods driven into the stream-bed or lake-bed and positioned well up in the euphotic zone of good light penetration (1 to 3 ft, or 0.3 to 0.9 m) for

maximum abundance and diversity of macroinvertebrates. A 4-ft (1.2 m) depth is acceptable unless the water is exceptionally turbid.

(2) The optimum period for substrate colonization is six weeks for most types of water. Three replicate samples at each station are an absolute minimum.

(3) For uniformity of depth, suspend the multiple-plate samplers from floats on ⅛-in. (3.2 mm) steel cable. If vandalism is a problem, use subsurface floats or put the sampler on supports placed on the bottom. Regardless of the installation technique, use uniform procedures (for example, the same depth and exposure period, sunlight, current velocity, and habitat type).

(4) At shallow water stations (less than 4-ft (1.2 m) deep), install samplers so that the exposure occurs midway in the water column at low flow. The samplers may be installed in pools or runs suspended below the water surface. The collections should be as representative of the reach as possible by ensuring that the samplers are not close to the bank.

(5) In streams up to a few metres in width, install the device at approximately midstream. In larger streams, install the device at approximately one-quarter of the total width from the nearest bank. Multiple-plate samplers may require additional weight for stability.

(6) If the samplers are installed in July when the water depth is approximately 4 ft (1.2 m), and the August average low flow is 2 ft (0.6 m), the correct installation depth in July is 1 ft (0.3 m) above the bottom. The sampler will receive sunlight at optimum depth 1 ft (0.3 m) and will not be exposed to air anytime during the sampling period. Care should be exercised not to allow the sampler to touch bottom which may permit siltation, thereby increasing the sampling error.

(7) In shallow streams with sheet rock bottoms, multiple-plate samplers can be secured to ⅜-in. (0.95 cm) steel rods that are driven into the substrate or secured to rods that are mounted on low, flat, rectangular blocks half-way between the water surface and the stream bed. However, these should be anchored securely to the rock bottom to avoid loss during floods.

(8) Factors such as the time of year and the body of water sampled should be considered in the determination of exposure time. The exposure time should be consistent among sites during the study. If study time limitation reduce this period, the data should be evaluated with caution, and in no case should data be compared from samplers exposed for different time periods.

(9) Samplers should be protected from loss of invertebrates during retrieval. Most insects rapidly leave the sampler when disturbed; thus a retrieval method to limit their escape should be used.

(10) In shallow water, approach the multiple-plate samplers from downstream, lift the sampler quickly, and place the entire sampler in a polyethylene bag or jug containing fixative. The fixative should be used only if the specimens collected require special processing for identification. Once the sampler is touched, it should be removed from the water immediately or many of the animals will leave the sampler. If the sampler should be disturbed during the recovery process so that it cannot be lifted straight up out of the water, a net should be used to enclose the sampler before it is disturbed.

(11) To accomplish this, the multiple-plate sampler should be enclosed either in a sieving bucket with U.S. Standard No. 30 sieve screen or by a dip net constructed of U.S. Standard No. 30 sieve or finer grit bolting cloth that can be pulled around the sampling device before retrieval. Also, samplers exposed in deep water may be enclosed in a retrieval net and brought to the surface by divers. If the sampler can be pulled quickly from the water without undue disturbance, it may not be necessary to enclose it.

(12) The organisms can be removed in the field by disassembling the sampler in a tub or bucket partially filled with water and scrubbing the plates with a soft-bristle brush to remove clinging organisms. The contents of the bucket are then poured through a No. 30 or 70 sieve and washed into a jar and preserved. If the organisms are not removed in the field, the multiple-plate samplers can be taken to the laboratory and disassembled if placed in a water-tight container or sturdy plastic bag containing a fixative or preservative. Also, due to its cylindrical configuration, the round multiple-plate sampler fits various wide mouth containers with tight lids for shipping and storage purposes. The samples should be labeled with the location, habitat, date, and time of collection.

(13) Cleaned multiple-plates can be reused to assemble multiple-plate samplers. Do not reuse the multiple-plates if there is reason to believe that they were exposed to contamination by toxicants (for example, chemicals or oils). These substances may be toxic to the macroinvertebrates or may inhibit colonization. Do not reuse the multiple-plates that have been exposed to fixatives or preservatives.

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SUMMARY OF CHANGES

The primary technical changes from the previous version of this standard (E1367-99) are summarized in this section.

(*1*) Information from USEPA (2001) (**1**) and Environment Canada (1994) were used to update the sections dealing with collection, storage, and manipulation of sediments.

(*2*) Information from the following standards were consolidated in Annex A1 (once this Annex has been approved, there will be a ballot started to with draw these 15 standards:

D4387-84 (2002) Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates

D4556-85 (2002) Guide for Selecting Stream-Net Sampling Devices for Collecting Benthic Macroinvertebrates

D4342-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ponar Grab Sampler

D4343-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler

D4344-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Smith-Mcintyre Grab Sampler

D4345-84 (1998) Practice for Collecting Benthic Macroinvertebrates with Van Veen Grab Sampler

D4346-84 (1997) Practice for Collecting Benthic Macroinvertebrates with Okean 50 Grab Sampler

D4347-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Shipek (Scoop) Grab Sampler

D4348-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Holme (Scoop) Grab Sampler

D4401-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Petersen Grab Sampler

D4407-84 (2002) Practice for Collecting Benthic Macroinvertebrates with Orange Peel Grab Sampler

D4557-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Surber And Related Type Samplers

D4558-85 (2002) Practice for Collecting Benthic Macroinvertebrates with Drift Net

E1468-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Basket Sampler

E1469-92 (2002) Practice for Collecting Benthic Macroinvertebrates with Multiplate Sampler