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Standard Guide for ~~Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing~~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 E 1391; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

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

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

1.1 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]

~~1.3Three documents, (Environment Canada~~ **and by Environment Canada (1994 (2), ~~USEPA~~) 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)**~~and Test Method E1706) provide supplemental guidance on procedures dealing with the collection, storage, characterization, and manipulation of sediments used in toxicological assessments.~~

~~1.4This guide is arranged as follows:~~). 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

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

[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.**

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 E 1706).

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.~~5~~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.

~~1.6The~~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.

~~1.7~~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 ~~limitations~~requirements prior to use.* Specific hazards statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

~~D1129Terminology Relating to Water~~ 1067 Test Methods for Acidity or Alkalinity of Water

~~D4387Classification of Grab Sampling Devices for Collecting Benthic Macroinvertebrates~~ 1126 Test Method for Hardness in Water

~~D4822Guide for Selection of Methods of Particle Size Analysis of Fluvial Sediments (Manual Methods)~~ 1129 Terminology Relating to Water

[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 ~~, Vol 11.05~~*, volume information, refer to the standard's Document Summary page on the ASTM website.

D4823Guide for Core-Sampling Submerged, Unconsolidated Sediments[5] 1426 Test Methods for Ammonia Nitrogen In Water
D 3976 Practice for Preparation of Sediment Samples for Chemical Analysis
D 4387 Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates
D 4822 Guide for Selection of Methods of Particle Size Analysis of Fluvial Sediments (Manual Methods)
D 4823 Guide for Core Sampling Submerged, Unconsolidated Sediments
E 729 Guide for Conducting Acute Toxicity Tests on Test Materials with Fishes, Macroinvertebrates, and Amphibians
E 943 Terminology Relating to Biological Effects and Environmental Fate
E 1241 Guide for Conducting Early Life-Stage Toxicity Tests with Fishes
E 1367Guide for Conducting 10-Day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods[4]
Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates
E 1525 Guide for Designing Biological Tests with Sediments
E 1611 Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids
E1383Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates[4]
1688 Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates
E 1706 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates
I EEE/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 E 729 and Test Method E 1706, Terminologies D 1129 and E 943, and Classification D 4387; for an explanation of units and symbols, refer to IEEE/ASTM SI 10.

4.Summary to Guide

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

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

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

6.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 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. ~~See Section 9 for additional information.~~ 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 ~~9 and 14~~10-14.

7.2 *Cleaning*—Test chambers and equipment used to collect and store sediment samples, prepare and store dilution water and stock solutions, and expose test organisms should be cleaned before use. New glassware and plasticware should be soaked in 1:1 concentrated acid prior to use. Soaking overnight is adequate for glassware. Soaking for seven days in HCl, followed by seven days in HNO_3, followed by seven days in deionized water is recommended for plasticware. Used sample containers should be washed following these steps: (—*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 E 1706). 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)* non-phosphate detergent wash, (*) the appointment of a laboratory health and safety officer with the responsibility and authority to develop and maintain a safety program, (2) triple water rinse, (*) the preparation of a formal, written health and safety plan, which is provided to each laboratory staff member, (3) water-miscible organic solvent wash (acetone followed by pesticide-grade hexane* **(4, 5)**, *(*) an ongoing training program on laboratory safety, and (4) water rinse, (5) acid wash (such as 5% concentrated hydrochloric acid), and (6) triple rinse with deionized-distilled water. Altering this cleaning procedure might result in problems. Many organic solvents might leave a film that is insoluble in water (Step 3). A dichromate-sulfuric acid cleaning solution can generally be used in place of both the organic solvent and the acid (Steps 3 through 5), but it might attack silicone adhesive. (See 9.10 for cleaning during sample collection.)*

8.Safety Hazards

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

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

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

~~9.Sampling and Transport~~

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

~~9.2Sediment collections have been made with grab and dredge sampling devices and core samplers (see Table 1 and Guide D4823). The advantages and disadvantages of the various collection methods have been reported previously~~

8.6 *Pollution Prevention, Waste Management, and Sample Disposal*—Guidelines for the handling and disposal of hazardous materials should be strictly followed (Guide D 4447). 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)** ~~and are summarized in Table 2. All sampling methods disturb the sediment integrity to a degree. It is important to obtain sediments with as little disruption as possible when using sediment toxicity evaluations for realistic laboratory evaluations of in situ conditions. Core sampling is preferred above other methods for this reason. Choosing the most appropriate sediment sampler for a study will depend on the sediment's characteristics, efficiency required, and study objectives. Several references are available that discuss the various collection devices **(9-13)**. Grab samplers can penetrate sediments to depths of 10 to 50 cm. Dredge samplers collect to a depth of 10 cm and disrupt sediment integrity. Core samplers collect up to 1 or 2 m when collected by hand or gravity. However, vibratory or piston corers can reach depths of 10 m. The depth of penetration is limited to 10 core diameters in sandy substrates and 20 diameters in predominately clay sediments. The efficiency of these samplers for benthic collections has been compared, and the grab samplers are less efficient collectors than the corers in general, but they are easier to handle in rough water, often require fewer personnel, and are obtained more easily~~); Fig. 1).

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

TABLE 1 2 Sampling Containers, Preservation Requirements, and Holding Times for Sediment SamplesA(EPA, 1296, 197). See also Rochon and Chevalier USEPA 2001(160))

Conventamional Sediment Variable	Container[B]		PreUservation
Acidity	P, G		Cool, 4°C
Acidity	Total organic carbon (TOC)		Cool, 4°C
Alkalinity	P, G		Cool, 4°C
Alkalinity	P, G		CNool, 4°C
AmmoniaP, G	Cool, 4°C	28 days	
ArmaP, G	Cool, 4°C	28 dizays	
Sulfate	P, G		Cool, 4°C
Sulfide	P, G		Co ol, 4°C
Sulfide	P, G		Con ol, 4°C
Sulfi te	P, G		Cool, 4°C
Sulf te	P, G		Cool, 4°C
Nitrate-nitrite	P, G		Cool, 4°C
Nitratncentrite	P, G		Cool, 4°C
Nitrite	P, G		Cool, 4°C
Nitite	P, G		Cool, 4°C
Oil and grease	G		C ool, 4°C
Oil anse	G		C of nol, 4°C
Organie	P, G		Cool, 4°C
			Metals[C]
Chromium VI	P, G		Cool, 4°C
Chromnium VI	P, G		Cool, 4°C
Mercury	P, G		
Mercury	P, G		
Metals (except Cr or Hg)	P, G		
Metable org)	P, G		
			Organic Compounds[C]
			Organic compounds[C]
Extractables (including phthalates, atrosamines organ ochlorine pe sticides, PCB's artroaromatics, isophorone, Polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons, and TCDD)			
Extractables (including phthalates, atrosamines Identification of appropriate reference sediments for biological tests artroaromatics, isophorone, Polynuclear aromatic hydrocarbons, haloethers, chlorinated hydrocarbons, and TCDD)			
G, PTFE-lined cap	Cool, 4°C	7 days (untilextraction) 30 days (after extraction)	
Acid Volatile Sulfide (AVS)	Cool, 4°C	Normalization of the concentrations of divalent metals sediments	
Extractractables (phenols)	G, PTFE-lined cap		Cool, 4°C
Extractractables (phenols)	Sediment grain size		Cool, 4°C
Purgables (hal ocarbon s and aromatics)	G, PTFE-linedseptum		Cool, 4°C
Purgables (hal of sediment toxics)	G, PTFE-lity testum		Cool, 4°C
Purga bles (acrolein and acrylonitrate)	G, PTFE-lined septum		Cool, 4°C

TABLE 2 1 Summary of Bottom Sampling EquipmentA(1))

Device	
PTFE or g**la sstubeShallow wadeable watersor deep w aters if SCUBA available. Soft orsemi-cons olida ted deposits.**	Pres erveslay sedimentdepo orlaboratory s
Clearly state the problem: purpose and objectives, available resources, members of the project team: For example, the purpose mights.	Pr be to evalu conditions, ev model. It is im
Hand corer with removable PTFE or glass liners	Same as abo obtained.
Box cor**er**	Same as abo
Ider	Samntify the
Gravity corers, that is, Phleger Corer	Deep lakes a
You**ng Grab (PTFE- or kynar-l ine d mod ified 0.1- m2 van Veen)** **Identify inputs to the decision: information and measuremen)**	Lakesa nd ma Lakets that ne
Ekman or box dredge	Soft to semis in waters of v
PONAR Grab Sampler	**Deep lakes, clay.**
PONAR Grab Sampler	**Define the st**
BMH-53 Piston Corer	Waters of 4 to semi-consolid
V **an VeenDee p lakes,r ivers, andestuaries. Useful on s and, silt, or clay.**	Adequ ateon permitting sub
Develop a decision rule: define parameters of interest and determine the value of a parameter thay.	At would caus exceedance c
BMH-60	Sampling mov
P**etersen Grab SamplerDeep lakes, rivers, andestuaries. Useful on mos tsu bst rates.**	Larges ample
Specify limits on decision errors: Establish the measurement quality objectives (MQOs) which include determining the level of confidence required from the data; precision, bids, repres.	Lentativeness
Shipek Grab Sampler	Used primarily reservoirs.
Orange-Peel Grab Smi th-McIntyre Grab	Deep l akes,ri
Optimize the Grab	D design: Cho select approp
Scoops, Drag Bucke t sVariousenvironments, depending on dep than dsubstrate.Inexpensive,e asy to hand le. Scian before the study begins regarding the sampling design (i.e., the frequency, number, and le.	Lossof fineso Location of fie objectives.

A Comments represent subjective evaluations.

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 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.

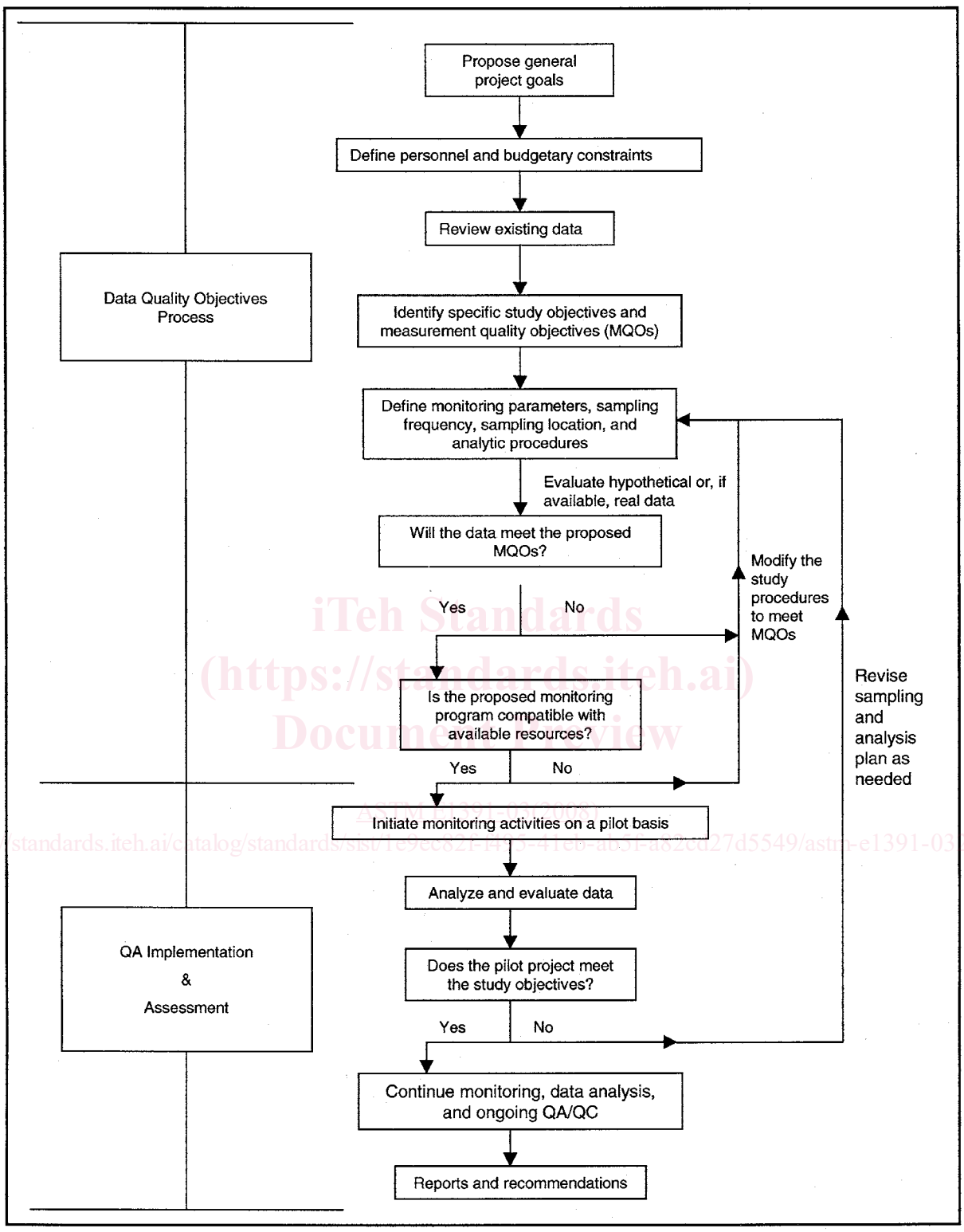

FIG. 1 Flow Chart Summarizing the Process that Should Be Implemented in Designing and Performing a Monitoring Study (modified from MacDonald et al. (1991 (10)); USEPA 2001 (1))

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

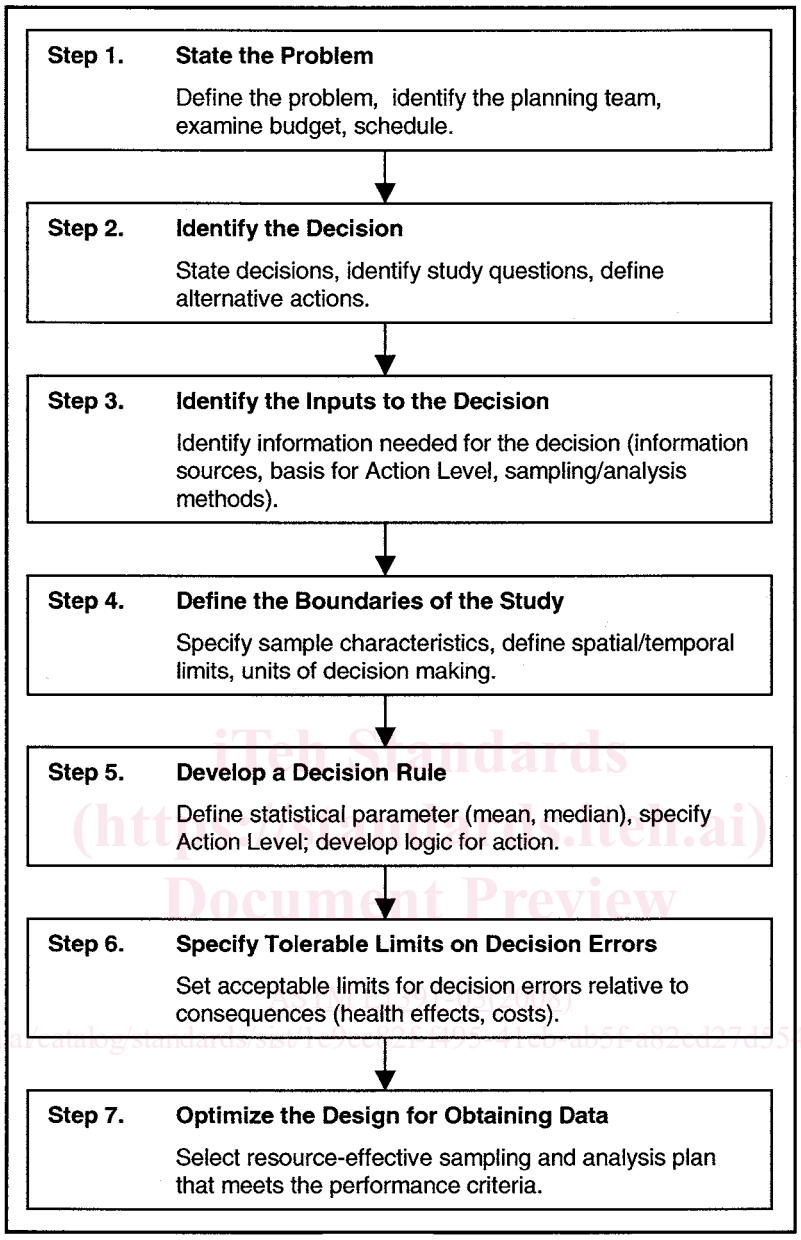

FIG. 2 Flow Chart Summarizing the Data Quality Objectives Process (after USEPA 2000a (12); 2001 (1))

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)**~~. Most of the reported studies used grab samplers, although box corers~~ **). 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 (~~15-17~~15)~~, gravity corers~~).

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 E 1706). 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 E 1706). 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)**~~, and hand collection~~ **; Barbour et al. 1999 (~~19-21~~19) ~~test methods are reported with increasing frequency.~~**

~~9.3The disadvantages of grab and dredge samplers (Table 2) include a shallow depth of penetration and the presence of a shock wave that results in loss of the fine surface sediments. Murray and Murray~~ **). 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)**~~, however, described a grab sampler usable in rough water that samples the top 1 cm of sediment quantitatively and retains fine materials. Other grab samplers that sample surface sediments quantitatively have been described by Grizzle~~) **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.

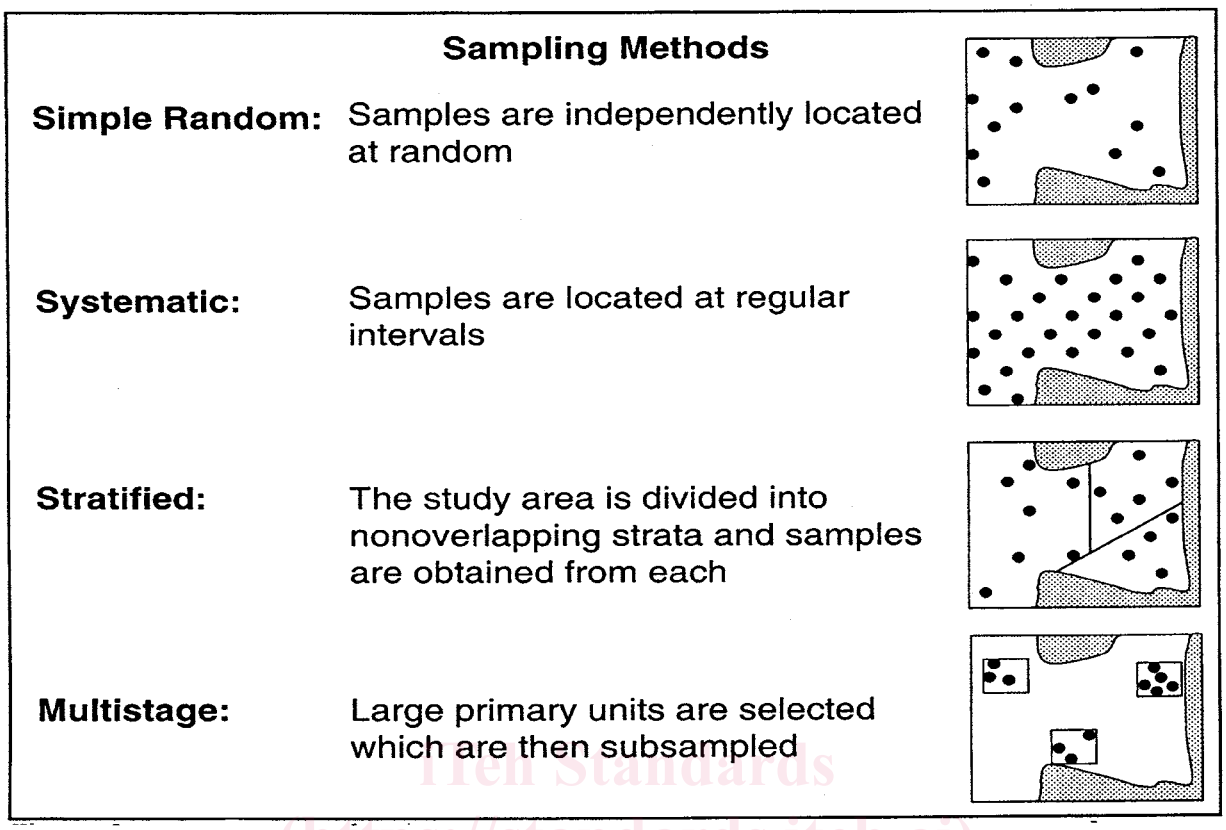

FIG. 3 Description of Various Sampling Methods (adapted from USEPA 2000c (20); 2001 (1))

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 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)**~~. The depth profile of the sample may be lost in removal of the sample from the sampler. Grab sampling promotes the loss of not only fine sediments (Table 2), but also water-soluble compounds and volatile organic compounds present in the sediment. Dredge samplers are appropriate only for collecting sediments that are to be dredged because they disrupt sediment integrity severely and lose surficial fines.~~

~~9.4Studies of macroinvertebrate sampling efficiency with various grab samplers have provided useful information for sampling in sediment toxicity and sediment quality evaluations. These data provide information that would indicate sampler efficiency at retaining surficial sediment layers. The modified van Veen is used commonly in coastal sampling~~ **, Strobel et al. 1995 (24)**~~. The Ekman grab is a commonly used sampler for benthic investigations (23). The Ekman's efficiency is limited to less compacted, fine-grained sediments, as are the corer samplers. Blomqvist~~ **) 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)** ~~reviewed the various Ekman modifications and their associated problems and concluded that the Ekman grab could be used reliably if caution was used during operation. The most commonly used corer is the Kajak-Brinkhurst corer. The Petersen, PONAR, and Smith-McIntyre grabs are used most often (~~). 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~~) in more resistant sediments. Based on studies of benthic macroinvertebrate populations the sediment corers are the most accurate samplers, in most cases followed by the Ekman grab~~ **). 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.

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 sediment; Klemm et al. 1990 **(30)**; GLNPO, 1994 **(11)**~~. The PONAR grab was the most accurate and the Petersen the least for compacted sediments~~ **; Long et al. 1996 (31); 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 (31)).**

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

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 **(35)**.

[C] Based on an average of 3 L of sediment per test chamber and 5 replicates (USEPA, 2000d **(35)**).