CHAPTER 4 *Field Sample Processing, Transport, and Storage of Sediments*

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/sediment system. Similarly, oxidation of anaerobic sediments increases the availability of certain metals (DiToro et al., 1990; Ankley et al., 1996). Materials and techniques should be selected to minimize sources of contamination and variation, and sample treatment prior to testing should be as consistent as possible.

A flowchart is presented in Figure 4-1 that summarizes common sediment processing procedures discussed in this section as well as issues and objectives relevant to each processing step.

4.1 Sample Containers

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.

Recommendation Box #1
Sample Containers

- ➡ High density polyethylene (HDPE) or polytetrafluoroethylene (PTFE) containers are suitable and preferred for most analytical measurements because they are made of relatively inert material and they are generally unbreakable.
- ➡ All containers should be pre-cleaned prior to filling with sample.
- ➡ Consider using certified, pre-cleaned containers, commercially available from many vendors.
- ➡ Purge containers with inert gas (e.g., nitrogen) prior to and after filling if anoxic conditions must be maintained.
- ➡ Fill containers completely if the sample will not be frozen prior to analysis.

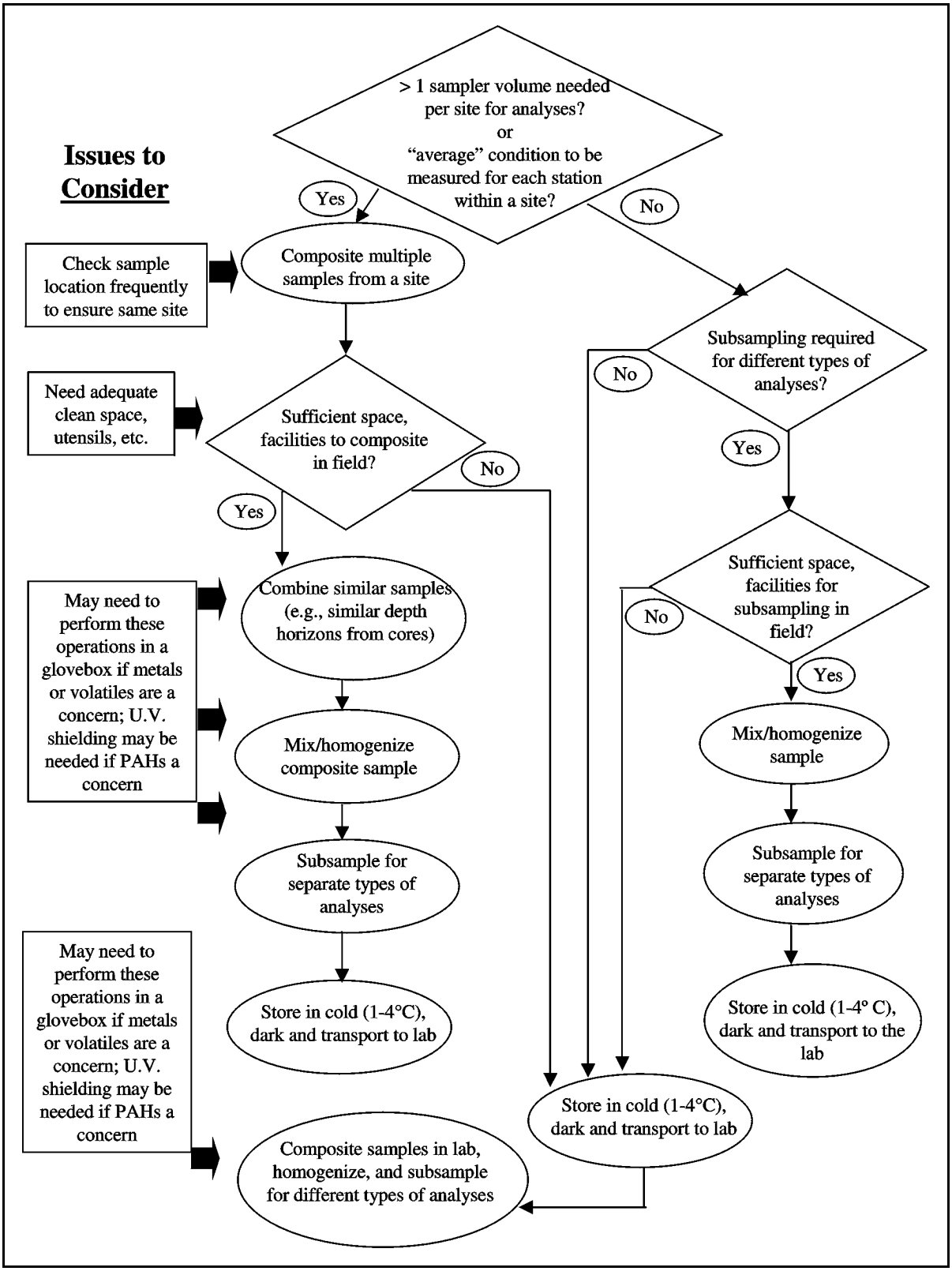

Figure 4-1. Flowchart of suggested sediment processing procedures

4.1.1 Container Material

Borosilicate Glass, and high-density polyethylene, polycarbonate and fluorocarbon plastics should be used whenever possible to minimize leaching, dissolution, and sorption (ASTM, 2000a; APHA, 1995). 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 4-1 summarizes the appropriate types of sampling containers and allowable holding times for various types of contaminants associated with sediments.

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 or Teflon®) 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 (e.g., clean aluminum foil). Plastic or acid-rinsed glass containers are recommended when the chemicals of concern are heavy metals.

4.1.2 Container Preparation

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.

If new containers are used, Environment Canada (1994) recommends that new glassware and plasticware should be soaked in 1:1 concentrated acid prior to use. Soaking overnight is adequate for glassware. For plasticware, the recommended procedure involves soaking for seven days in hydrochloric acid (HCl), followed by seven days in nitric acid (HNO_3), followed by seven days in deionized water. Shorter soaking times might be satisfactory in most instances (ASTM, 2000a). Used sample containers should be washed following these steps: (1) non-phosphate detergent wash, (2) triple water rinse, (3) water-miscible organic solvent wash (acetone followed by pesticide-grade hexane), (4) water rinse, (5) acid wash (such as 5% concentrated HCl) and (6) triple rinse with deionized-distilled water. 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 any silicone adhesive present in the container. See ASTM (2000a) and USEPA (2000d) for further information.

If a sample is to be refrigerated, the container should be filled to the brim to reduce oxygen exposure. This is particularly critical for volatile compounds (e.g., AVS). If a sample is to be frozen, the container should be filled to approximately 90% of its volume (i.e., 10% headspace) to allow for expansion of the sample during freezing. See Section 4.4 for preservation and storage conditions for various types of analyses. For studies in which it is critical to maintain the collected sediment under anoxic conditions (e.g., where metals are the pollutants of concern), the container should be purged with an inert gas (e.g., nitrogen) before filling and then again before capping tightly.

Table 4-1. Recommended sampling containers, holding times, and storage conditions for common types of sediment analyses (USEPA, 1983;1993; ASTM, 2000a). 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*	R, dark
Bioaccumulation testing	P, PTFE	2 weeks*	R, dark

*Holding time might be longer depending on the magnitude and type of contaminants present. See Section 4.5.

All sediment containers should be properly labeled with a waterproof marker prior to sampling. Containers should be labeled on their sides in addition to or instead of labeling the lids. Each label should include, at a minimum, the study title, station location and/or sample identification, date and time of collection, sample type, and name of collector. Blind sample labeling (i.e., a sample code) should be used, along with a sample log that identifies information about each sample (see Section 2.7) 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 Section 2.7 and 7.6). Labeled containers should be stabilized in an upright position in the transport or storage container (see Section 4.4 Transport and Storage for further information). Extra containers should be carried on each sampling trip.

4.2 Subsampling and Compositing Samples

The decision to subsample and/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 and/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 available, or alternatively, in a laboratory setting following sample transport.

Recommendation Box #2

How should sediment samples be subsampled and composited?

- ➡ 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 Teflon®, 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 hours (and preferably within 24 hours) 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.

4.2.1 General Procedures

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.

Compositing refers to combining aliquots from two or more samples and analyzing the resulting pooled sample (Keith, 1993). Compositing is often necessary when a relatively large amount of sediment must be obtained at 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 (see Table 2-2), but not to dilute a polluted 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).

All utensils (e.g., spoons, scoops, spatulas) which come in direct contact with sediment samples during handling and processing should be made of non-contaminating materials (e.g., glass, high-quality stainless steel and/or Teflon®).

Considerations

All handling procedures carry the risk of sample contamination. Therefore, sediment sample handling should be kept to a minimum. Potential sample contamination can be caused by the following common situations...

- making field measurements of sediments using contaminated probes, utensils, or other instruments.
- contaminated and uncontaminated stations are sampled without appropriate decontamination of equipment between stations.
- the parameter of interest is volatile (e.g., ammonia, acid volatile sulfides, or volatile organics) and samples are exposed to air.
- samples are exposed to vessel exhaust fumes, lubricants, or rust.

4.2.2 Grab Samples

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 Section 4.1 for recommendations on containers). The container must be large enough to accommodate the sediment sample and should be tightly sealed with the air excluded.

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 Section 3.1.1 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 (e.g., Ponar, VanVeen).

Prior to 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; PSEP, 1997a). If the overlying water in a sediment sampler is turbid, it should be allowed to settle if possible.

Considerations

When working with grab samples...

- decanting the water, or opening the jaws lightly to let the water run out is not recommended as these methods might result in unacceptable disturbance or loss of fine-grained sediment and organic matter.
- if metal contamination or sediment oxygen demand are of concern, oxidation of sediments could significantly alter their characteristics. Process the sample in a glovebox or similar apparatus under an oxygen-free environment.
- for samples that are suspected of heavily elevated polynuclear aromatic hydrocarbons (PAHs), process immediately under low light upon retrieval to minimize ultraviolet light-activated toxicity of PAHs (Ankley et al., 1994).

The general subsampling and compositing process for grab samples is illustrated in Figure 4-2. Subsampling can be performed using a spoon or scoop made of inert, non-contaminating material. *Sediment which 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 approximately 10% head space in the container to accommodate expansion and avoid breakage.

There are two alternatives for compositing sediment samples from grab samplers (see Figure 4-2): (1) compositing and homogenizing (mixing) in the field and (2) compositing in the field and homogenizing in the laboratory.

In some studies (e.g., where metals are the pollutants of concern), it might be necessary to subsample a grab sample under oxygen-free conditions to minimize oxidative changes. In these cases, it is recommended that a hand-coring device 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, 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) or very important (Besser et al., 1995), depending on the sediment characteristics, the contaminants of concern, and the study objectives.

Checklist
Compositing samples involves

In the field

- ✓ placing subsamples from individual grab samples in a clean container to form a composite sample
- ✓ transporting the composite to a laboratory
- ✓ homogenizing the sample at the laboratory to prepare it for testing (See Section 4.3 for further details)

In the lab

- ✓ placing subsamples from multiple grabs in a clean container
- ✓ mixing the subsamples to form a homogeneous composite sample
- ✓ placing the composite sample in one or more containers, depending on the number of analyses to be performed
- ✓ transporting the composite sample to a laboratory (or laboratories) for testing

4.2.3 Core Samples

Subsampling sediment core samples is usually done to focus the assessment on a particular sediment horizon or horizons and/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.

Systematic subsampling (see Figure 4-3) 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 (e.g., 25-50 cm) are typically used to characterize a site.

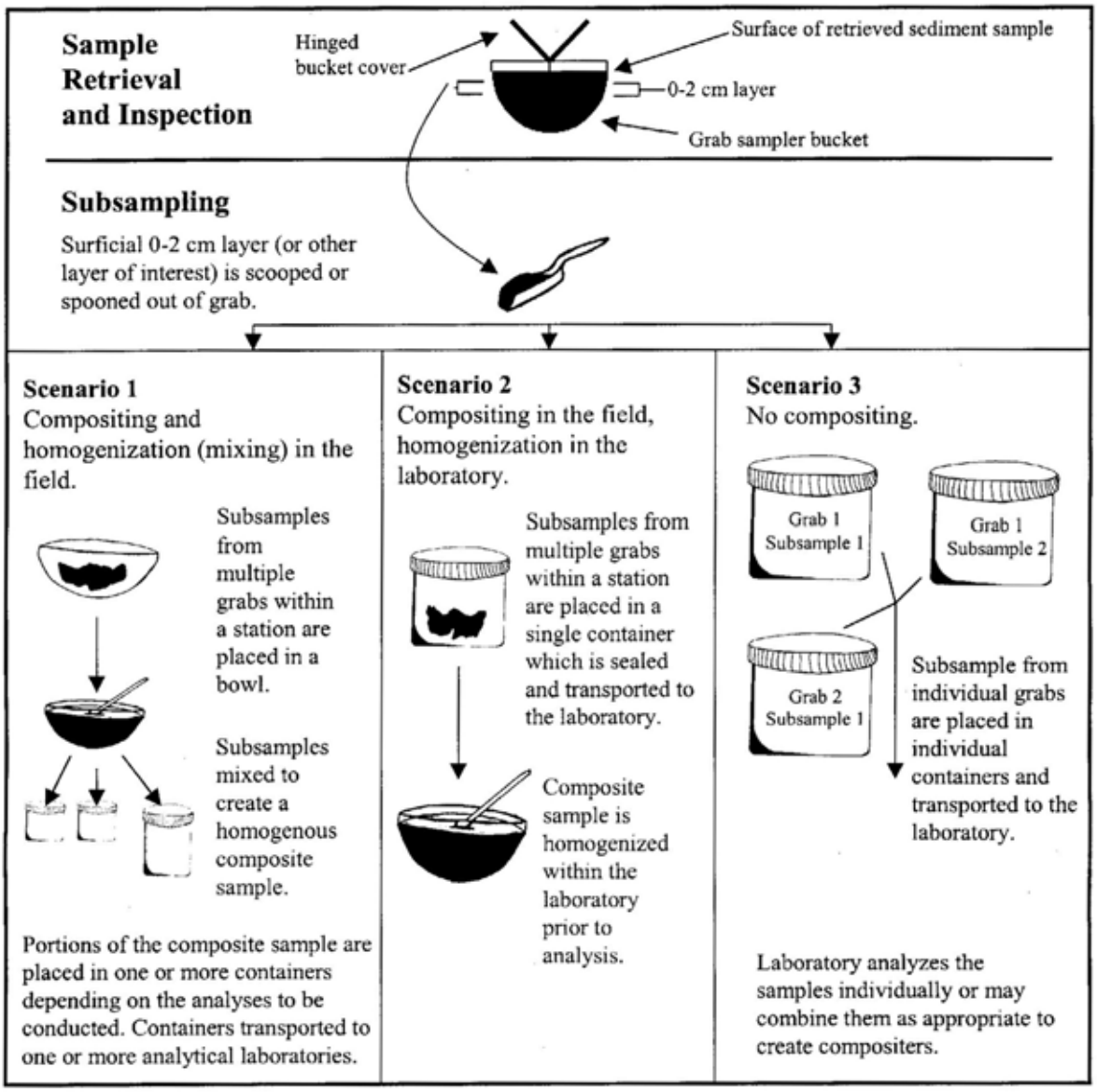

Figure 4-2. Alternatives for subsampling and compositing sediment grab samples.

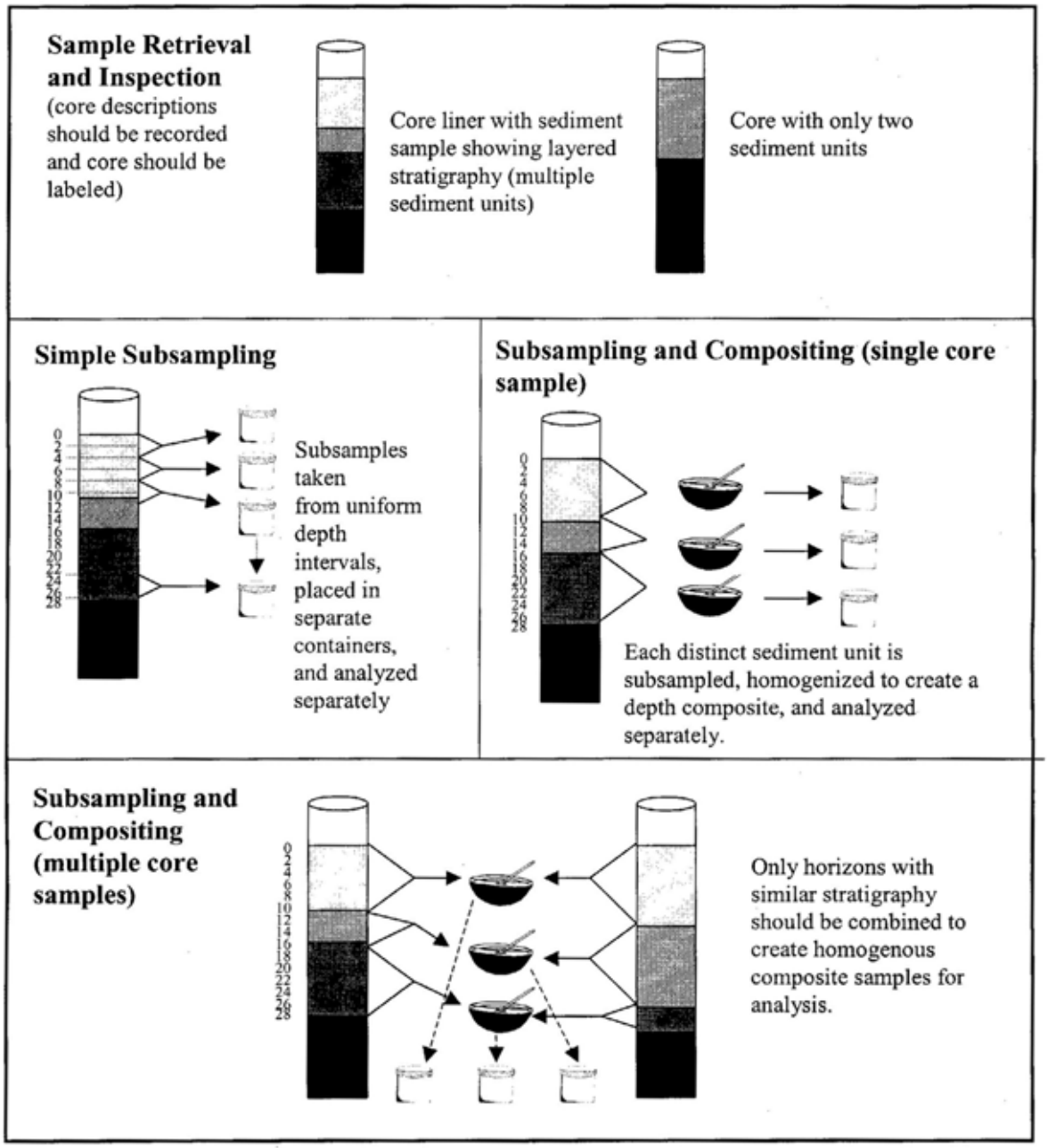

Figure 4-3. Alternatives for subsampling and compositing sediment core samples.

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 might be important factors determining which core sections are sampled. In these studies, subsampling depth intervals include the 0 to 2 cm layer (for recent deposition) and the 0 to 5 cm or 0 to 15 cm layers (for biological activity, depending on resident organisms). Many programs have project-specific depths corresponding to study requirements, such as dredging depths for navigation or remediation dredging. In many regional or national environmental monitoring programs (e.g., EMAP), the uppermost surficial layer is sampled because information on the horizontal distribution of sediment contaminants is desired (USEPA, 2000d).

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 teflon or nylon string.

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). [Note: For dredged material studies and other types of remediation projects, where pre-determined depth strata are not necessarily defined, it is usually important to view the entire core prior to sectioning or compositing.] 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 avoid 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 Teflon® cutter (Environment Canada, 1994; Mudroch and Azcue, 1995). 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; Mudroch and MacKnight, 1994).

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 avoid contamination of the sediment by liner material, and the core itself can be sliced with Teflon® or nylon string. The core then becomes two D-shaped halves that can be easily inspected and subsampled (Mudroch and Azcue, 1995). 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).

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 they must be used with care. Spoons or

scoops have also been used to subsample surface sediments from a box corer (Environment Canada, 1994).

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, it is recommended that only horizons of similar stratigraphy 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 (Figure 4-3). Composite samples must be homogenized prior to analysis or testing.

4.3 Homogenization

Homogenization refers to the complete mixing of sediment to obtain consistency of physicochemical properties throughout the sample prior to 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.

4.3.1 General Procedures

Prior to homogenization, unrepresentative materials (e.g., twigs, shells, leaves, stones, wood chips and seagrass) are often removed and documented in an appropriate field log (see Section 5.2 for techniques to remove unrepresentative material). The need for removal of larger matter depends on the analyses to be conducted.

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; Stemmer et al., 1990a;b). This can alter the bioavailability of contaminants, particularly metals, by increasing or decreasing their availability (Ankley et al., 1996). If metal contaminants or volatile chemicals are a concern, samples should be mixed in a glovebox under an inert atmosphere and quickly partitioned into sample containers for analysis.

Recommendation Box #3
How should samples be homogenized?

- Use a sufficiently large, precleaned glass or stainless steel mixing bowl to homogenize the sample.
- Use clean glass polyethylene, or stainless steel implements (e.g., 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 (Burton et al., 1989; Ingersoll and Nelson, 1990; Johns et al., 1991a; Carr and Chapman, 1992).
- Regardless of the mixing method selected, the effectiveness of the method should be demonstrated using a homogenate replicate.

Photo courtesy of Nile Kemble

Homogenizing a composited sediment sample using a mechanical mixer

Photo courtesy of Chris Ingersoll

Subsampling sediment for toxicity testing

Mixing should be performed in a large, precleaned glass or stainless steel bowl. The sediment should be thoroughly stirred with a clean glass, high density polyethylene, or stainless steel spoon until textural, color, and moisture homogeneity are achieved (Environment Canada, 1994; PSEP, 1995). Hand mixing has also been performed by rolling the sediment out flat on a sheet of plastic or pre-combusted foil and tumbling the sediment by alternately raising each corner of the sheet (Mudroch and Macknight, 1994). This procedure, however, is not recommended where the anaerobic integrity of the sediment must be maintained.

Considerations
Homogenization of anaerobic sediments...

! Beware of over-mixing and/or introducing air to the sample. Such mixing is likely to change the chemical characteristics of the sample and yield unrepresentative results. This is especially important if samples are initially anaerobic or if volatile or labile chemicals are of interest (e.g., AVS).

Mechanical mixers have also been used to homogenize samples (Ditsworth et al., 1990; Stemmer et al., 1990b; Kemble et al., 1993), including portable cement mixers (bare metal and Teflon-lined) and portable drills fitted with a variety of stainless steel paddles (Kemble et al., 1994b).

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. After the sediment has been homogenized, it is generally partitioned among sample containers. Partitioning sediments for chemical or toxicity analyses 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 is 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 (ASTM, 2000a). An alternative is to use a splitter box designed to contain and then divide the homogenized sediment.

4.4 Sample Transport and Storage

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

4.4.1 General Procedures

Proper storage conditions (see Table 4-1) should be achieved as quickly as possible after sampling. For those parameters that are preserved via refrigeration (e.g., toxicity) 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. For samples that can be preserved via freezing (e.g., some metal and organic chemical analyses) dry ice can be used to freeze samples for temporary storage and transport (USEPA, 1983, 1993). Pelletized dry ice has been used effectively in the dredged materials management program to store core samples. It is important to know chilling capacities and

efficiencies to assure 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.

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 may transport samples to the laboratory themselves or utilize an overnight courier service. If a freight carrier is employed, the user must be aware of any potentially limiting regulations (e.g. regarding the use of ice or dry ice). Samples that have a recommended storage temperature should be cooled to that temperature prior to placement in the transport container. Light should be excluded from the transport container.

Core samples should be transported as intact core liners (tubes). Prior to 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 (e.g., 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). 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 Section 4.3.3).

Impregnating unconsolidated sediment cores with epoxy or polyester resins will preserve sediment structure and texture (Ginsburg et al., 1966; Crevello et al., 1981) but not sediment chemical characteristics. Therefore, this procedure is not recommended for transporting or storing sediment samples for chemical characterization or biological testing (Environment Canada, 1994).

Recommendation Box #4
Sample Transport and Storage

- ➡ The volume of overlying water in sediment samples should be minimized to reduce the potential for resuspension of surface sediments during transport.
- ➡ Care should be taken to retain the surficial floc overlying a core sample.
- ➡ Cores should be secured in an upright position during transport to minimize disturbance of the sediment.
- ➡ Sediment samples collected in the field should be stored in containers without headspace at 4° C and in the dark to minimize changes in contaminant bioavailability.
- ➡ Prior to transport, headspace in the core liner should be filled with site water and both ends of the liner should be completely sealed.

4.5 Sample Holding Times

Limits for effective holding times are governed by sediment type and contaminant characteristics (ASTM, 2000a). Because these qualities are not always known, a general recommendation is to store sediments and interstitial water in the dark at 4 °C (SETAC, 2001). Preservation and recommended storage times for various types of analyses are summarized in Table 4-1.

Recommendation Box #5
How long should samples be stored before analysis?

- Unless site-specific information is available, sediment samples should be stored no longer than two weeks prior to using in toxicity testing.
- Preserved samples for chemical analyses should be stored no longer than the maximum holding times as defined by the particular program.

Samples collected for toxicity tests should be used as quickly as possible. Recommended maximum holding times range from 10 days (NOAA) to two weeks (ASTM, 2000a; USEPA, 2000d), to eight weeks (USEPA/ACOE, 1991, 1998). Preferred sample storage times reported for toxicity tests have varied substantially (Dillon et al., 1994; Becker and Ginn, 1990; Carr and Chapman, 1992; Moore et al., 1996; Sarda and Burton, 1995; Sijm et al., 1997; Defoe and Ankley, 1998), and differences appear to depend primarily upon the type or class of contaminant(s) present.

Extended storage of sediments that contain high concentrations of labile contaminants (e.g., ammonia, volatile organics) might lead to loss of these contaminants and a corresponding reduction in toxicity. Under these circumstances, the sediment should be tested as soon as possible after collection, but not later than two weeks (Sarda and Burton, 1995). Sediments that exhibit low to moderate toxicity might exhibit higher variability in toxicity when tested following storage of short duration (e.g. two weeks). Testing could actually be more reliable following longer storage for these types of samples if the longer storage reduces potential interference associated with indigenous predators (DeFoe and Ankley, 1998). Sediments contaminated with relatively stable compounds (e.g. high molecular weight compounds such as PCBs) or those that exhibit moderate-to-high toxicity, do not seem to vary appreciably in toxicity with increased storage time (Moore et al., 1996; DeFoe and Ankley, 1998). Longer term storage might be acceptable in such cases. Given our incomplete knowledge on the changes that occur, it is recommended that sediments should be stored no longer than two weeks for toxicity testing unless site-specific information indicates otherwise.

Periodic measurements of contaminants of concern provide a useful context for interpretation of toxicity test results when sediments or interstitial waters are stored for extended periods of time, but this is rarely cost-effective. It might be more efficient to conduct interstitial water toxicity tests within two weeks of sediment collection, corresponding with the start of sediment tests (Ingersoll et al., 1993). In general, though, interstitial water should be analyzed as quickly as possible following sampling to minimize possible changes in contaminant bioavailability.

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).